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FURTHER DEVELOPMENTS OF THE END QUENCH HARDENABILITY TEST

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Abstract

A hardenability test bar selectively quenched from both ends has been developed to provide correlations with the thermal histories of quenched plates that correspond more closely than is possible with the standard single end quench specimen. For those steels where the pearlitic reaction may be avoided but which transform partially or wholly under the same cooling conditions to acicular structures of the bainite type, this test should be useful in predicting behavior at the section center, since the mechanical properties at the center of symmetrical double end quench specimens may be determined. Experimental cooling curves are reported and a chart of specimen dimensions versus the thickness of plates quenched in agitated water is included.

WITHIN recent years, metallurgical application of the single end quench test originated by Jominy (1) to (7)¹ has become of increasing interest for the evaluation of hardenability as affected by variations of analysis, of grain size, and of mass.

The single end quench test supplies excellent and duplicable information concerning response to heat treatment under limited conditions of heat transfer. It is usually possible to establish a posi-

¹The figures appearing in parentheses pertain to the references appended to this paper.

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tion at a given distance from the end of this test bar whose hardness will correspond in practice to the center *hardness*, for example, of any quenched section. *Unless* the cooling of this particular point is sufficiently rapid to develop maximum hardness, or *unless* it corresponds closely to that of the selected section—there is no assurance that identical metallographic structures have been achieved.

It is well known that, where the maximum mechanical properties of a particular steel in a given hardness range may be attained by tempering a martensitic structure, increasing the section size (i. e., decreasing the quenching rate) to an extent that will allow partial transformation to *ferrite* or increasing the alloy content to a degree that will inhibit the *full austenite transformation* during quenching and tempering, will usually result in inferior ductility, impact, and other test values. Since, as will be shown, a selected position of the single end quench test bar [for which the cooling rate at 1300 degrees Fahr. (705 degrees Cent.) corresponds to that at the center of a quenched plate] will have a different cooling rate in a critical transformation zone below 1000 degrees Fahr. (540 degrees Cent.) for some deep hardening steels, and that even this rate will be changed by compositional variations (Fig. 7), it is evident that the present test possesses limitations.

Appreciation of the importance of relating cooling or quenching rates to the transformation characteristics portrayed by the Bain S-curves has suggested the development of an end quenching technique to accomplish the reliable and convenient reproduction of plate cooling curves, without resorting to full-scale sections, and which would also furnish a transverse central zone of uniform thermal history susceptible to mechanical and metallographic examination. It was proposed, therefore, that the simple expedient of quenching the test bar simultaneously from *both* ends, as the length, diameter and perhaps shape were changed, would permit the establishment of a family of cooling curves from which those corresponding to particular sections could be selected for laboratory investigation. It was recognized that, while variations of composition would unquestionably change the shapes of the test bar cooling curves according to the temperatures at which partial or complete transformation occurred, it was probable that, once the *dimensions* of the *double end quench test bar* had been established to approximate the cooling of the section under investigation *for a constant analysis, a general equation* for the time-temperature relationships between section and

test bar size would follow that would be independent of composition.

Experimental evidence supporting this hypothesis is presented in this paper, which is the initial report of an extended investigation of the relation of hardenability, quenching rate, and section size to the mechanical properties of cast alloy steels after various heat treatments.

EXPERIMENTAL PROCEDURE

Although it is usually possible to select a point at some distance from the water-cooled end of the 4-inch single end quench test bar

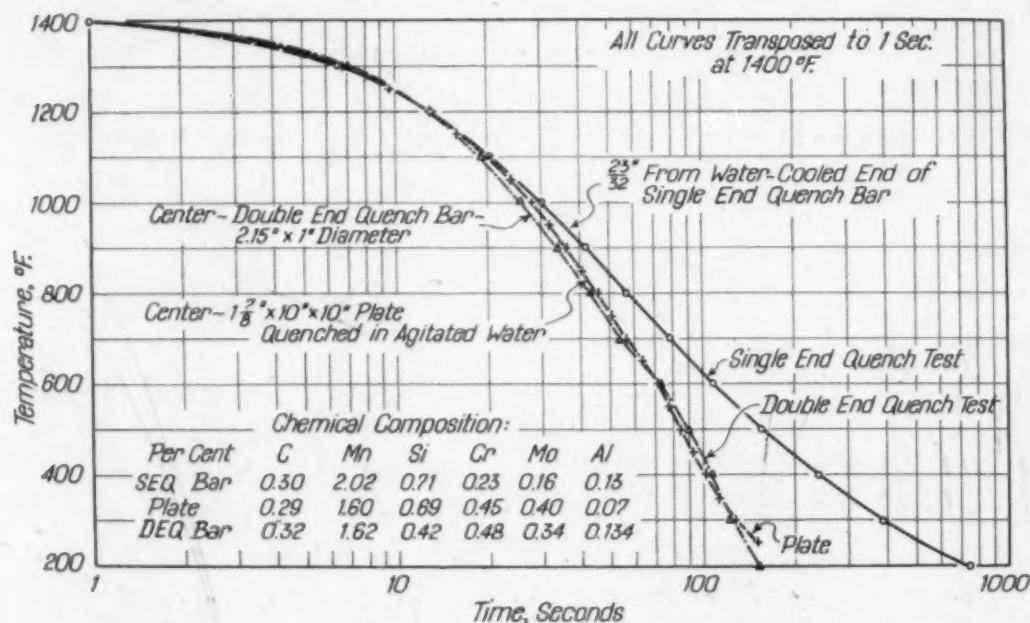
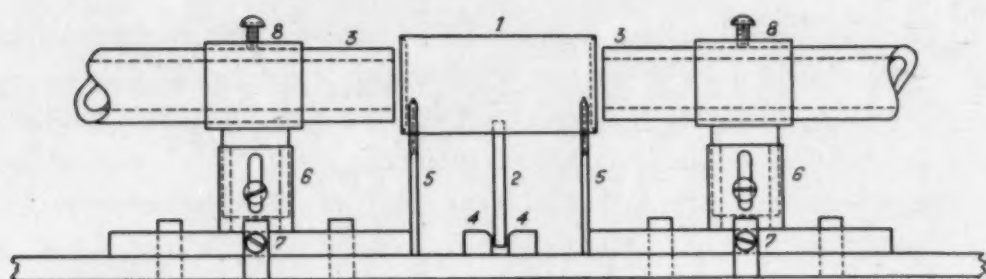


Fig. 1—Comparison of Plate with Single and Double End Quench Cooling Curves. Quenched from 1650 Degrees Fahr.

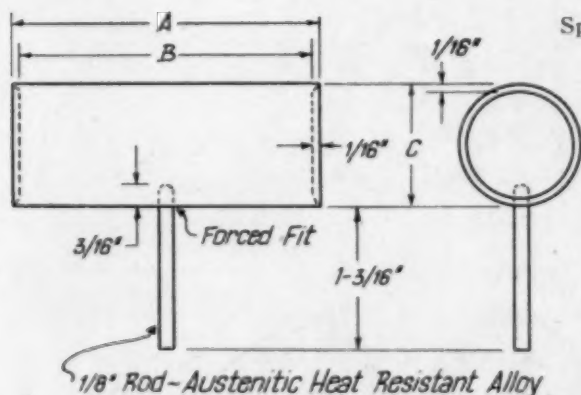
that has the same cooling rate at a designated temperature as a given location in any other shape or section, it is also evident that the cooling curves at reference points of metal sections quenched from one face and from two or more faces will be different. To illustrate, a point $\frac{23}{32}$ inch along the single end quench specimen has the same cooling rate at 1300 degrees Fahr. (705 degrees Cent.) as the center of a $1\frac{7}{8}$ by 10 by 10-inch plate, quenched in agitated water (Fig. 1). The test bar, however, cools progressively more slowly in the lower temperature ranges. The total time to cool from 1400 to 250 degrees Fahr. (760 to 120 degrees Cent.) exceeds that of the plate by $6\frac{1}{2}$ minutes.

Since plate quenching is characterized by heat loss from two



- 1-Double End Quench Specimen
 3- $\frac{1}{2}$ " Diameter Orifice
 5-V-Knife Edge Specimen Support
 6-Vertical Adjustment to Accommodate Specimens of Different Diameters
 7-Horizontal Adjustment to Accommodate Specimens of Different Lengths
 8- -- -- of Location of Orifice
- 2-Centering Pin
 4-Centering Slot

Fig. 2—Double End Quenching Fixture.



Specimen Sizes Employed in Determining Families of Cooling Curves:

A	B	C
1 $\frac{1}{4}$ "	1 $\frac{1}{8}$ "	1"
2"	1 $\frac{3}{8}$ "	1"
2 $\frac{1}{2}$ "	2 $\frac{1}{8}$ "	1"
2 $\frac{1}{2}$ "	2 $\frac{3}{8}$ "	2"
4"	3 $\frac{1}{8}$ "	1"
6"	5 $\frac{1}{8}$ "	1 $\frac{1}{2}$ "
8"	7 $\frac{1}{8}$ "	2"

Fig. 3—Double End Quench Specimen.

directions, it was logical to consider cooling the hardenability bar from both ends. Initial efforts to accomplish this demonstrated the superiority of a horizontal specimen over one held vertically during quenching. When it became apparent that the cooling at the $\frac{23}{32}$ -inch point of the 4-inch specimen could not be sufficiently modified to conform to that of the $1\frac{7}{8}$ -inch plate below 1000 degrees Fahr. (540 degrees Cent.), shorter test bars were tried. Further experiments showed that the *center* cooling curves of double end quench specimens and of plates were of similar form, and it became desirable to investigate the extent of this correlation.

The modified quenching fixture (Fig. 2), in which the test bar rests on two V-knife edges, may be adjusted to accommodate the several specimen sizes. Rate of water flow, from a 50-gallon tank overhead, is regulated to 450 cubic inches per minute, equivalent to a linear velocity of 194 feet per minute, from each $\frac{1}{2}$ -inch diameter

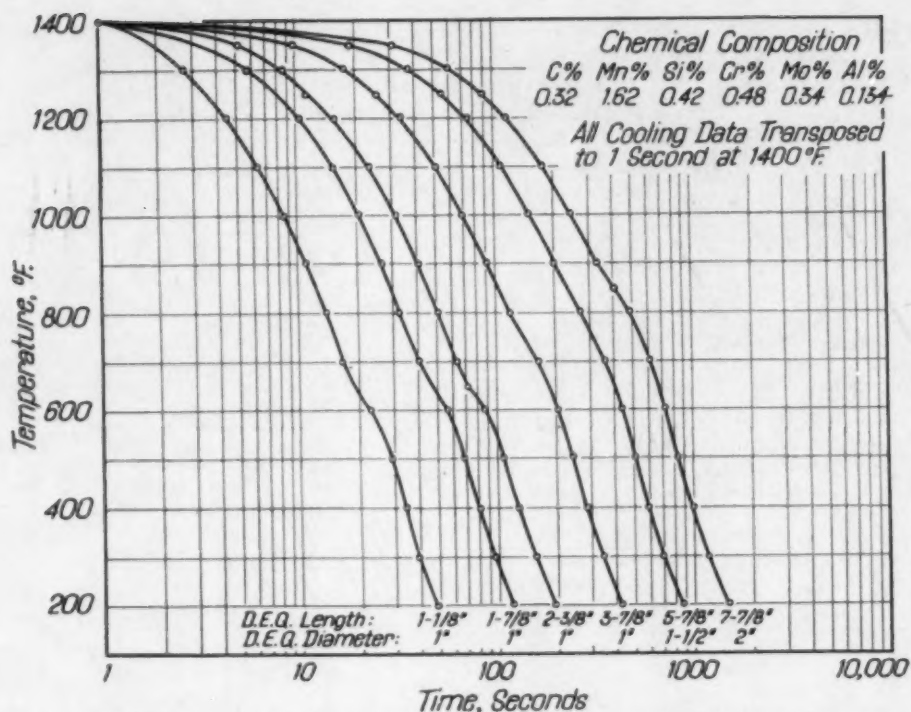


Fig. 4—Center Cooling Curves ($\frac{1}{32}$ Inch Below Surface) for Double End Quench Test Bars of a Manganese-Chromium-Molybdenum Steel, Quenched from 1650 Degrees Fahr.

orifice. The water temperature is maintained at 75 ± 5 degrees Fahr.

The cupped ends of the test bar (Fig. 3) confine the quenching to these faces. A small center pin to facilitate handling serves also to orient the specimen in the quenching unit. To avoid scaling during heating, heat resistant alloy cups, containing a thin graphite disk, are placed over each end of the specimen.

The test bar, thus protected, is charged into a hot furnace, austenitized at the selected temperature for the required time, transferred as quickly as possible to the fixture, and quenched simultaneously at both ends.

COOLING CURVES

To determine the cooling curves, a No. 22-gage chromel-alumel thermocouple was welded to the specimen at the selected location and the test bar quenched as described. A Leeds and Northrup "Speedo-max" recorder furnished a continuous graph of cooling from which the time-temperature data were plotted in more convenient form.

Cooling curves have been obtained at the center ($\frac{1}{32}$ inch below the surface) of double end quench specimens² $1\frac{1}{8}$ to $7\frac{7}{8}$ inches long

²Distance between quenched faces is employed to identify double end quench test bar length. Overall length including "cups" is $\frac{1}{8}$ inch greater.

Table I
Double End Quench Tests
Reproducibility of Cooling Data

Specimens Quenched from 1650 Degrees Fahr. (Mn-Cr-Mo Cast Alloy Steel)								
Temp. °F.	D.E.Q.— $3\frac{7}{8}$ " x 1" Diameter				D.E.Q.— $5\frac{7}{8}$ " x $1\frac{1}{2}$ " Diameter			
	(1)	(2)	(3)	(4)	(1)	(2)	(3)	(4)
1400	45	35.5	36.5	35	54.0	60.0	50.0	53.0
1300	61	53.0	51.7	52.6	89	94	87	92
1200	78	68.7	68.5	69	128	126	122	128
1100	96	85	86.2	85.9	161	166	155	163
1000	113	104	102	105	199	204	195	205
900	136	126	126	127	250	253	244	255
800	164	154	153	157	334	329	323	327
700	210	203	206	203	424	420	406	412
600	255	244	243	243	494	487	472	482
500	290	278	281	281	563	559	543	551
400	338	321	321	318	655	646	623	638
300	384	376	376	373	765	759	740	743
200	470	458	462	456	941	930	910	930
Cooling data transposed to 1 second at 1400 degrees Fahr.								
1400	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1300	17	18.5	16.2	18.6	36	35	38	40
1200	34	34.2	33	35.0	75	67	73	76
1100	52	51	50.7	51.9	108	107	106	111
1000	69	70	67	71	146	145	146	153
900	92	92	91	92	197	194	195	203
800	120	120	118	122	281	270	274	275
700	166	169	171	168	371	361	357	360
600	211	210	208	208	441	428	423	430
500	246	244	246	246	510	500	494	499
400	294	287	286	283	602	587	574	586
300	340	342	341	338	712	700	691	691
200	426	424	427	421	888	871	861	878

Table IA
Center Cross Sectional Hardness Exploration

Double End Quench—7 $\frac{7}{8}$ " x 2" Diameter Specimens									
Heat No.	Chemical Composition—Per Cent								
	C	Mn	P	S	Si	Ni	Cr	Mo	Al
1	0.31	0.82	0.028	0.035	0.55	0.05	0.10	0.36	0.038
2	0.31	1.12	0.025	0.030	0.62	0.05	0.32	0.37	0.047
3	0.32	1.29	0.026	0.029	0.60	0.08	0.44	0.38	0.053
4	0.31	1.75	0.029	0.031	0.68	0.07	0.58	0.41	0.053
Transverse Distance from Center Inches	Heat No.								
	1	2	3	4	Hardness—Rc Scale				
$\frac{31}{8}$ "	18.5	33.0	37.5	42.5					
$\frac{3}{4}$ "	18.0	35.0	38.0	43.0					
$\frac{5}{8}$ "	19.0	34.5	38.5	45.0					
$\frac{1}{2}$ "	19.5	33.5	38.0	47.0					
$\frac{3}{8}$ "	18.5	33.5	36.0	46.0					
$\frac{1}{4}$ "	17.5	34.0	37.0	45.5					
$\frac{1}{8}$ "	19.0	32.5	37.0	45.0					
0	18.0	33.0	37.0	45.0					
$\frac{1}{8}$ "	18.5	34.5	37.5	44.0					
$\frac{1}{4}$ "	19.0	33.5	38.0	43.0					
$\frac{3}{8}$ "	20.0	34.0	39.0	43.0					
$\frac{1}{2}$ "	17.0	33.5	39.5	43.0					
$\frac{3}{4}$ "	19.5	33.0	40.0					
$\frac{1}{4}$ "	19.0					
Avg. Rc	18.7	33.7	38.0	44.5					

* $\frac{31}{8}$ " reading taken on longitudinal flat.

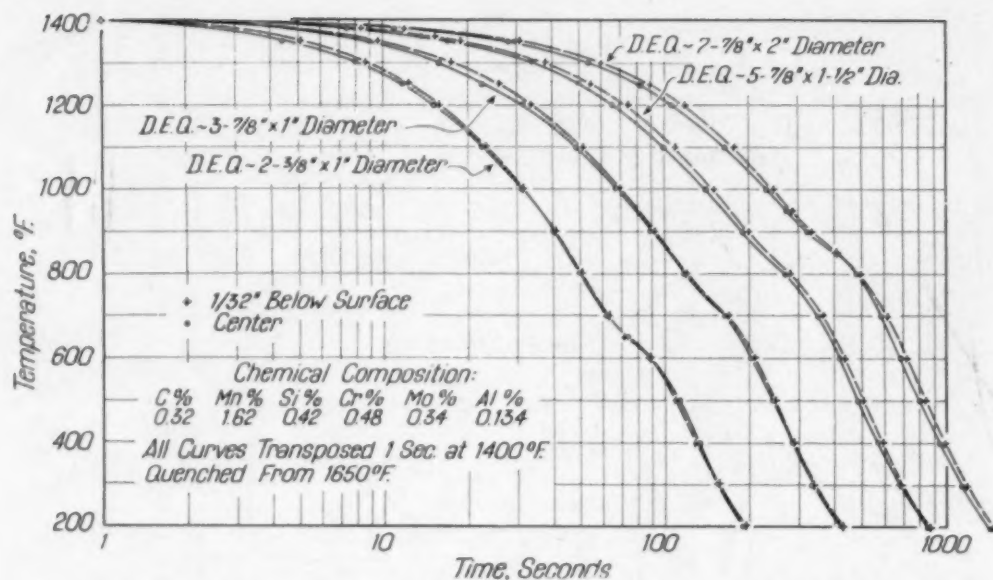


Fig. 4A—Comparison of Center and Surface Cooling Curves Midway Between Ends of Double End Quench Specimens of a Manganese-Chromium-Molybdenum Steel.

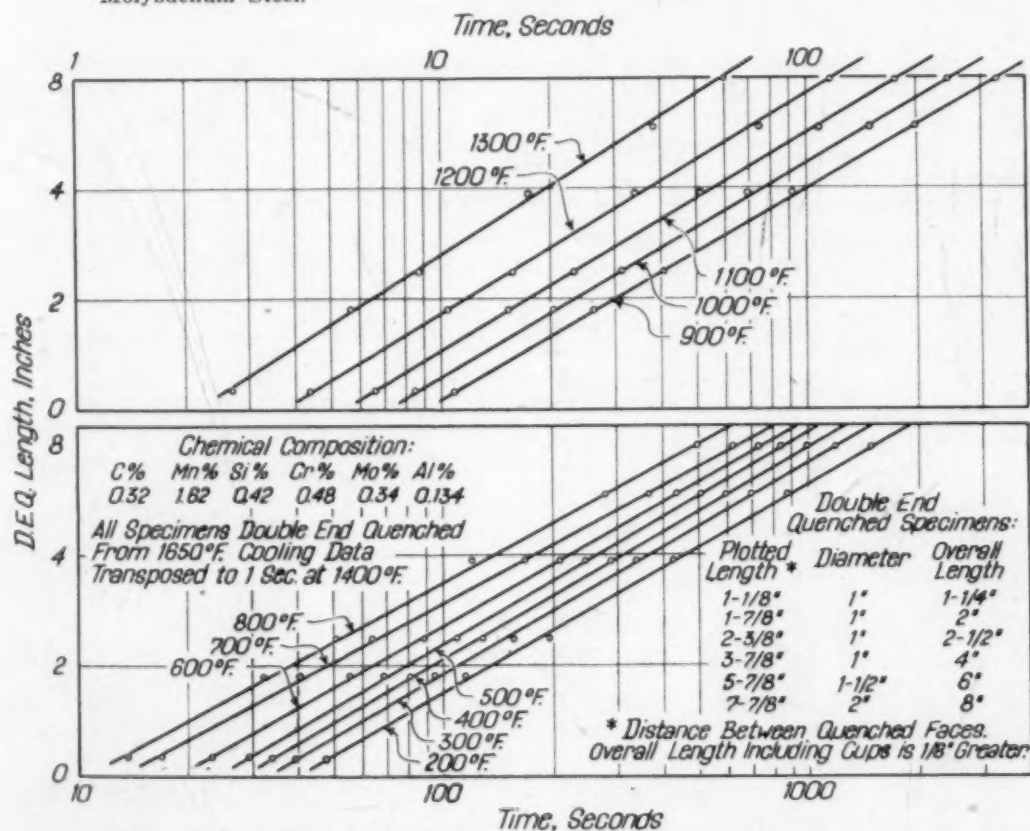


Fig. 5—Time to Cool at Center ($\frac{1}{32}$ Inch Below Surface) of Specimen to Indicated Temperature Versus Length of D.E.Q. Test for a Manganese-Chromium-Molybdenum Cast Steel.

of a Mn-Cr-Mo steel (Fig. 4). While each plot represents the average of several experiments, the excellent reproducibility of in-

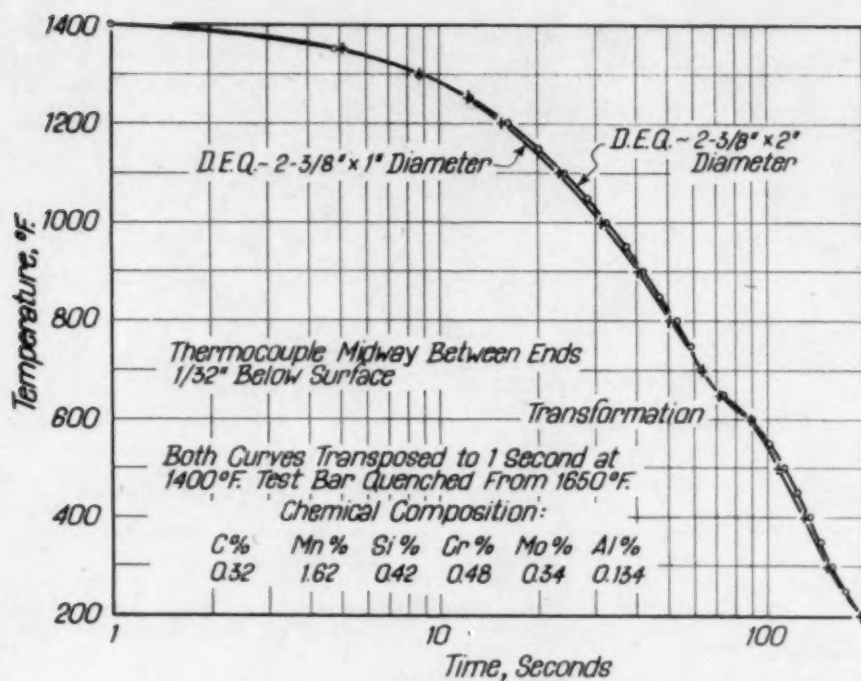


Fig. 6—Effect of Diameter on Cooling 2 $\frac{3}{8}$ -Inch Double End Quench Bar.

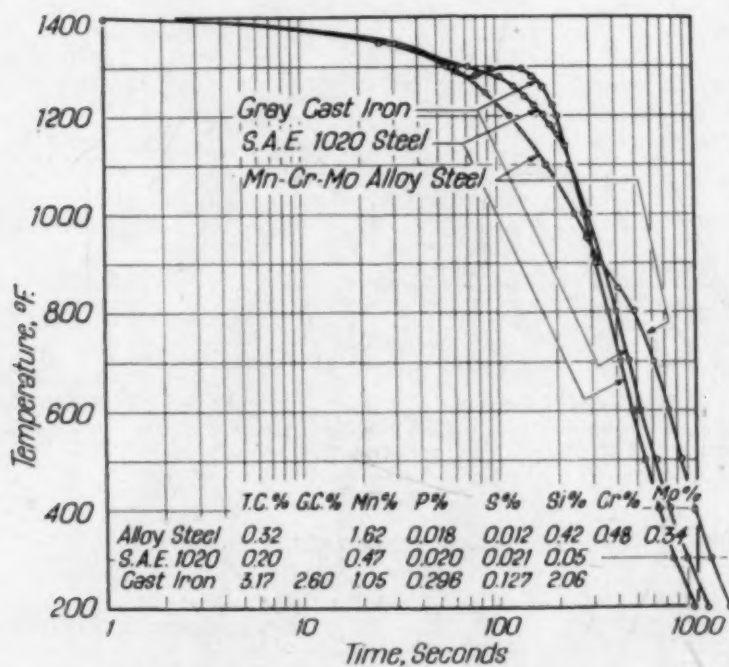


Fig. 7—Effect of Composition Upon Center Cooling Curve for 7/8-Inch Double End Quench Test Bar. All Curves Transposed to 1 Second at 1400 Degrees Fahr. Quenched from 1650 Degrees Fahr.

dividual determinations is evident in Table I. To eliminate slight variations in the time-temperature data caused by differences in the

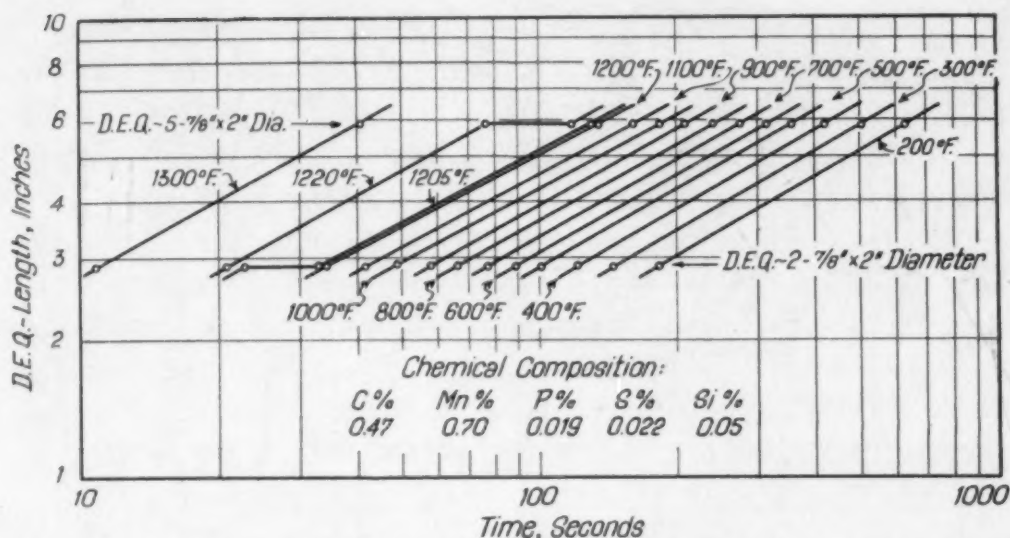


Fig. 8—Time to Cool at Center ($\frac{1}{32}$ Inch Below Surface) of Specimen to Indicated Temperature Versus Length of Double End Quench Test Bars of S.A.E. 1045 Steel. All cooling data transposed to 1 second at 1400 degrees Fahr. Test bars quenched from 1650 degrees Fahr.

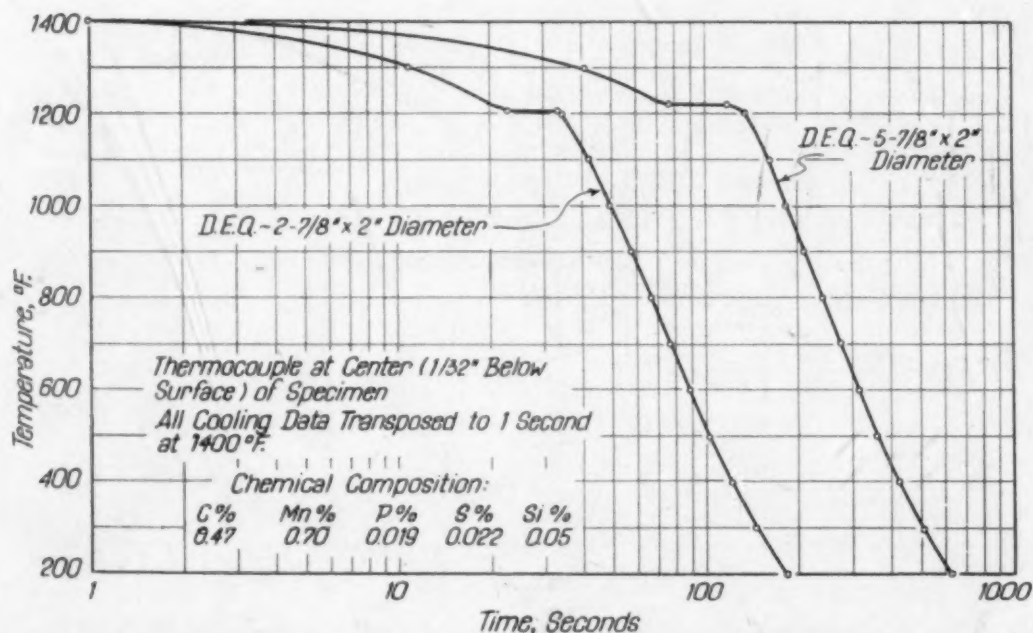


Fig. 9—Center Cooling Curves for $2\frac{7}{8}$ -Inch and $5\frac{7}{8}$ -Inch Specimens of S.A.E. 1045 Steel Double End Quenched from 1650 Degrees Fahr.

time of transfer from furnace to quench, 1400 degrees Fahr. (760 degrees Cent.) was chosen as an arbitrary point of reference above the Ar' range. The cross sectional uniformity of cooling rates at the longitudinal center of the several double end quench specimens is demonstrated by the center and surface ($\frac{1}{32}$ inch below) cooling curves in Fig. 4A. The transverse hardness surveys of Table 1A

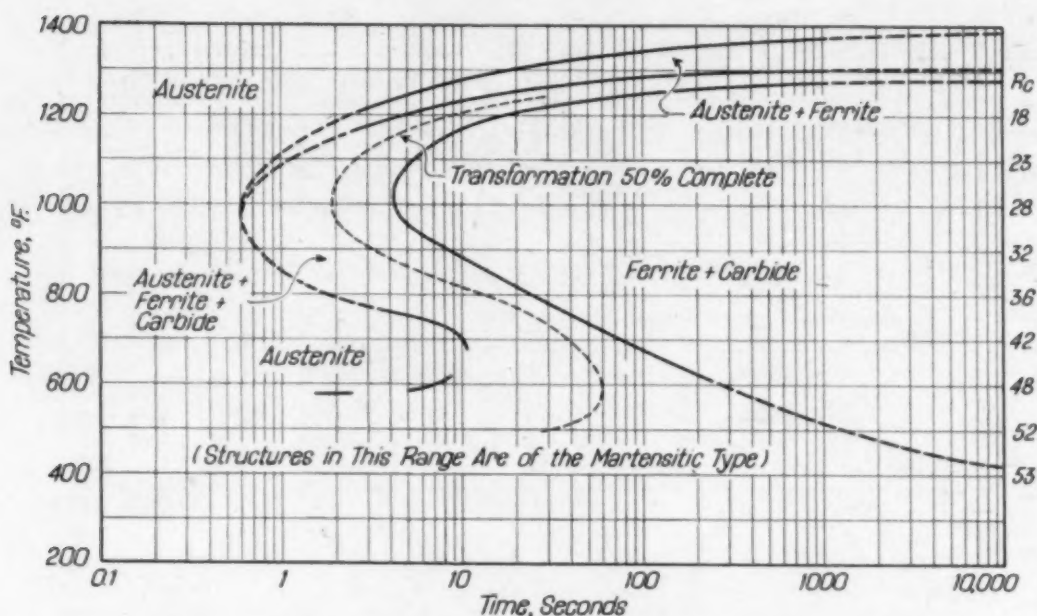


Fig. 10—Isothermal Transformation Diagram for S.A.E. 1050 Steel. (0.50 Per Cent Carbon, 0.91 Per Cent Manganese). Austenitized at 1670 Degrees Fahr.*

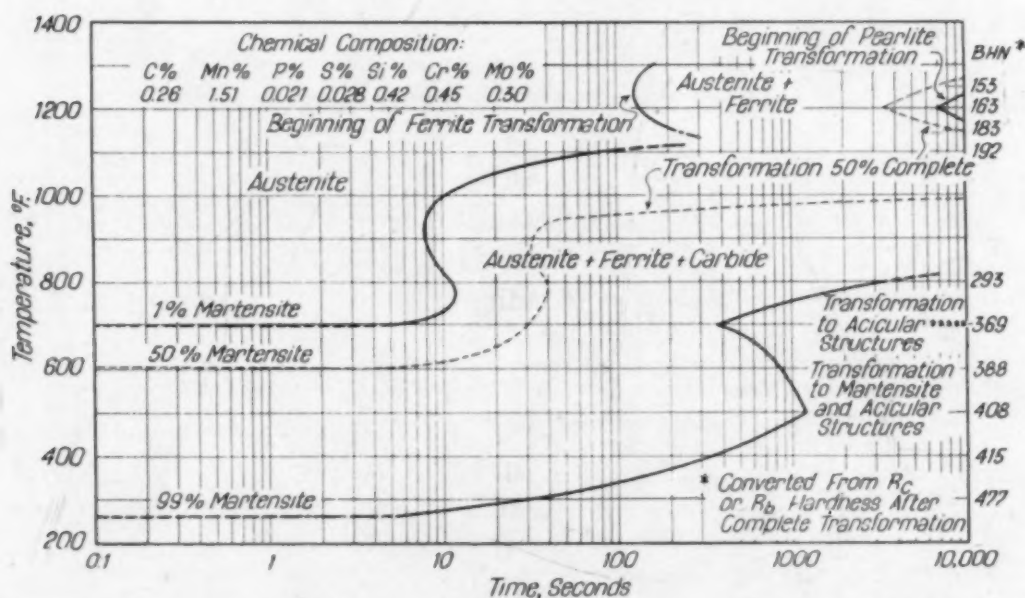


Fig. 11—Isothermal Transformation Diagram for a Manganese-Chromium-Molybdenum Steel. Austenitized at 1650 Degrees Fahr.

at the center of $7\frac{7}{8}$ -inch test bars double end quenched from 1650 degrees Fahr. offer further confirmation with changing hardenability.

For this Mn-Cr-Mo steel, an essentially linear logarithmic plot (Fig. 5) of time to cool to a designated temperature from 1400 degrees Fahr. versus length of the double end quench test bar obtains from $1\frac{1}{8}$ to $3\frac{7}{8}$ inches, on 1-inch diameter specimens. To extend

*United States Steel Corporation, "Atlas of Isothermal Transformation Diagrams."

this relationship, it was necessary to increase the diameters of the $5\frac{7}{8}$ and $7\frac{7}{8}$ -inch tests to $1\frac{1}{2}$ and 2 inches respectively, indicating that a maximum length-diameter ratio of approximately 4:1 should be observed to avoid extraneous radial cooling effects. The close correspondence of the center cooling curves (Fig. 6) from $2\frac{3}{8}$ -inch test bars of 1 and 2-inch diameters indicates that the minimum ratio is not critical.

From Fig. 5, the center cooling curve for any double end quench test bar length, within the range of the available data on this particular Mn-Cr-Mo steel, can be interpolated. That the matching of cooling curves cannot be expected if different alloys are involved is illustrated by a comparison of an alloy steel, a carbon steel, and gray cast iron (Fig. 7).

The essentially complete linearity in Fig. 5 for this Mn-Cr-Mo steel is a fortunate convenience, but it should not be construed as a general relationship for all compositions. For comparison, a similar graph has been prepared for S.A.E. 1045 steel (Fig. 8). The original cooling curves appear in Fig. 9. The linearity obtains here only before and after complete transformation.

The "S" curves in Figs. 10 and 11 are typical of these two steels. Although they are based on isothermal quenching, they will serve to explain the basic cooling differences in Figs. 4 and 9, since the reaction curves for a continuous or finite cooling rate would lie below, and to the right of, the corresponding curves on the isothermal diagram.

The S.A.E. 1045 steel, when quenched from the austenitic condition, will begin to transform above 1000 degrees Fahr. (540 degrees Cent.) unless cooled very rapidly. The reaction itself is fast and will be manifested by the familiar arrest in the cooling curve, which results in a definite break in the linear time-temperature-length relationship in Fig. 8. If this steel is cooled quickly enough to avoid the pearlitic reaction, no transformation is expected until the martensite point is reached.

The transformation of the Mn-Cr-Mo steel (Fig. 11) is easily avoided above 1000 degrees Fahr. (540 degrees Cent.), except for very slow quenching rates. While rapid cooling is necessary to obtain a *completely* martensitic structure since transformation starts relatively quickly below 1000 degrees Fahr. (540 degrees Cent.), the reaction rate is sufficiently slow that only a small but observable modification is noted in the cooling curve. In more conventional

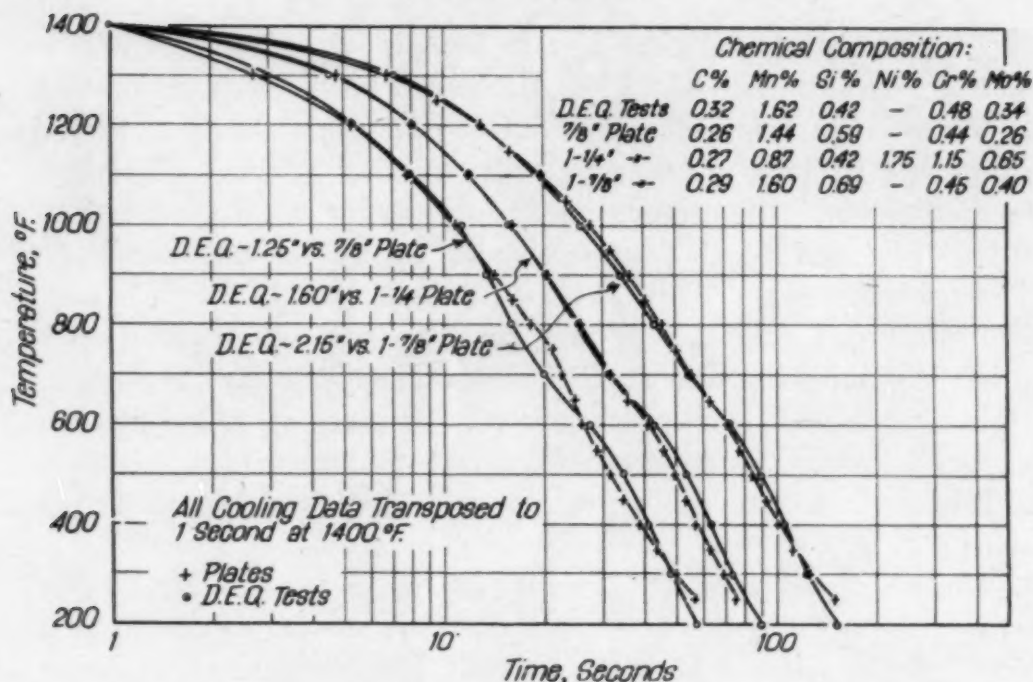


Fig. 12—Comparison of Cooling Curves at the Center of Plates Quenched in Agitated Water with those of Selected Double End Quench Specimens. Quenched from 1650 Degrees Fahr.

terms, the heat of transformation has been released at lower temperatures and distributed over a wider range because of the alloy content of the more complex steel.

CORRELATION WITH PLATE COOLING CURVES

The center cooling curves for $\frac{7}{8}$, $1\frac{1}{4}$, and $1\frac{7}{8}$ -inch plates, quenched in agitated water, are compared with those for selected double end quench specimens in Fig. 12. Since heavy scaling introduced serious variables, all plates were sand blasted prior to each heat treatment.

The *times* of cooling at the center of the several plate sections (Fig. 12) from 1400 to 1200 degrees Fahr. (760 to 650 degrees Cent.) ($\frac{7}{8}$ -inch, 4.3 seconds; $1\frac{1}{4}$ -inch, 7.0 seconds; $1\frac{7}{8}$ -inch, 12 seconds) were applied to the 1200-degree Fahr. line of Fig. 5 to establish the double end quench test *length* corresponding to each section. The interpolated center cooling curves for 1.25, 1.60 and 2.15-inch test specimens were then plotted in Fig. 12 together with those of the respective plates.

Since composition largely controls the conditions of heat evolution caused by transformation during cooling, it is essential, for

purposes of strict correlation, that the comparisons be made on the same material. The plate cooling curves (Fig. 12) for deep hardening material comparable to that of the test bars, but not of identical alloys, will suffice however to demonstrate the nature of the agreement until more adequate data are available. Since the rate of cooling above the temperatures for transformation is governed by the heat conductivity of the material, by the section size and by the quenching conditions, the close correspondence of the plate and test bar cooling curves above 700 to 900 degrees Fahr. (370 to 480 degrees Cent.) in Fig. 12 is excellent.

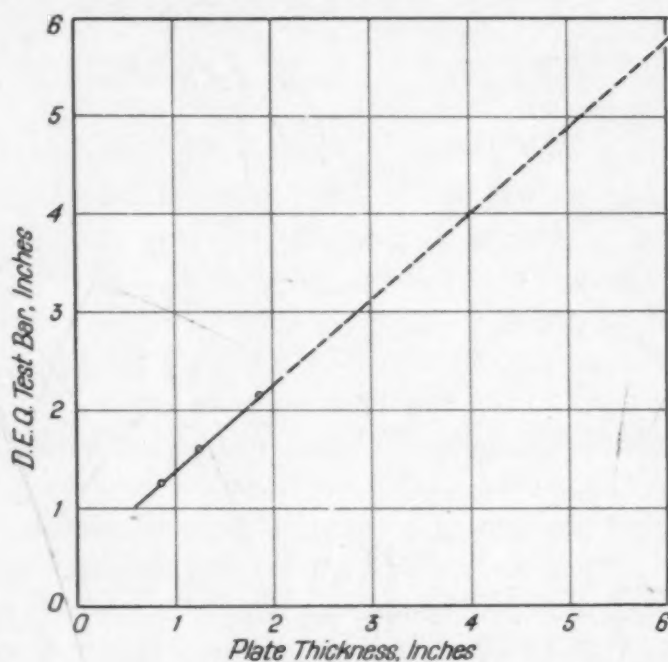


Fig. 13—Relationship Between Plate Thickness and Double End Quench Test Bar Length for Plates Quenched in Agitated Water. Length-diameter ratio of 4:1 must not be exceeded for double end quench test bars.

While transformation is occurring, other variables in addition to composition, such as heterogeneity from segregation, grain size, and distribution of nucleating centers, will likely modify the cooling characteristics. It would probably be necessary, for complete matching of the thermal histories, to obtain the double end quench specimens not only from the same material but from the same plates with which they are to be compared. The advantages and limitations of this requirement are discussed later.

In comparison, the obvious deviation from the plate cooling curve of the corresponding location ($\frac{23}{32}$ inch from the water-cooled

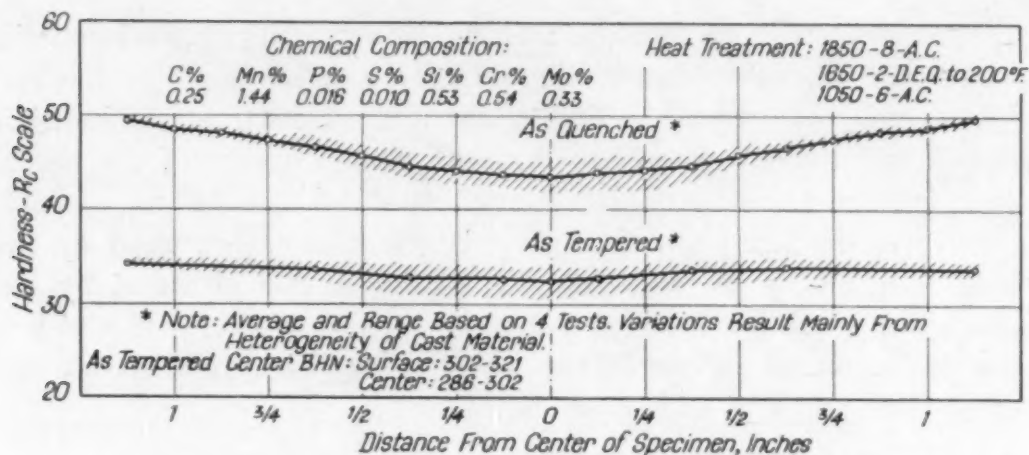


Fig. 14—As-Quenched and As-Tempered Hardness Surveys for DEQ—2 $\frac{3}{8}$ Inch by 2-Inch Diameter Specimens of a Manganese-Chromium-Molybdenum Steel.

end) on the single end quench was previously indicated (Fig. 1). The divergence in cooling in the lower temperature ranges may be critical for those deeper hardening steels whose transformation is easily avoided above 1000 degrees Fahr. (540 degrees Cent.) but which transform partially or completely under the same cooling conditions to acicular products below 900 degrees Fahr.

While the shapes of the cooling curves are affected by chemistry, the relationship between double end quench *test bar length* and *plate thickness* (Fig. 13), for a designated quench, is considered to be independent of composition, although this must be further examined empirically. To extend the present correlation, the determination of the center cooling curves for heavier plates is currently planned. Comparison with similar data from other investigations not yet released for publication indicates that this curve may be extrapolated with confidence for simple as well as for alloy steels.

The present double end quench test bar was developed specifically to match the thermal histories at the center of flat plates. The correlation for other sections has not been determined. The possible modifications in the shape of the test bar and/or of the quenching technique which might permit similar correlations with other types of sections may be such as to limit the broader applications of the technique.

REPRODUCIBILITY OF MECHANICAL PROPERTIES

Although the double end quench test bar center cooling data (Table I) exhibited excellent reproducibility, this was further in-

Table II
Mechanical Properties

Comparison of Immersion Quench and Double End Quench for Similar Center Hardness,
As-Quenched
(All Specimens Obtained from 2¼" Diameter x 10" Bars)

Chemical Composition—Per Cent						
C	Mn	P	S	Si	Cr	Mo
0.25	1.44	0.016	0.010	0.53	0.54	0.33
	Yield Strength* Psi.	Ult. Tens. Strength Psi.	% Elong. in 2"		% Red. Area	Hard- ness BHN
	Immersion Quenched 1½" x 1½" x 4¼" Bars ^(a)					
Average of 8 tests	138,780	148,884	16.4		48.9	321
Minimum	136,500	146,070	15.5		47.5	321
Maximum	142,000	151,750	17.5		50.6	321
	D.E.Q.—2¾" x 2" Diameter Test Bars ^(b)					
			% Elong. in 1"			
Average of 8 tests	130,320	143,695	16.8		51.4	302
Minimum	128,000	141,600	16.0		49.5	286
Maximum	132,800	146,700	18.0		53.9	321

Note: (a) Heat Treatment: 1850 Degrees Fahr.—8 Hours—Air Cool
1650 Degrees Fahr.—2 Hours—Oil Quench: 42-43 Rc Center
1050 Degrees Fahr.—6 Hours—Air Cool
.505" diameter tensile specimens—2.0" gage length

(b) Heat Treatment: 1850 Degrees Fahr.—8 Hours—Air Cool
1650 Degrees Fahr.—2 Hours—D.E.Q. to 200 Degrees Fahr.:
42-44 Rc Center
1050 Degrees Fahr.—6 Hours—Air Cool
.252" diameter tensile specimens—1.0" gage length

*Drop of Beam.

vestigated with respect to: 1. As-quenched hardness; 2. As-tempered hardness; 3. Mechanical properties.

Four 2¼-inch diameter by 10-inch bars, heated over the full length, were poured from the same heat of Mn-Cr-Mo steel. After homogenizing (1850 degrees Fahr.—8 hours—air cool), each bar was sectioned to provide two double end quench 2¾ by 2-inch diameter specimens from one half, and two 1½ by 1½ by 4¼-inch samples from the other. The latter received a normal heat treatment (full immersion oil quench from the hardening temperature followed by tempering for 6 hours), while the hardenability bars were double end quenched to 200 degrees Fahr. Four of these, from alternate positions in the original cast bars, were tempered immediately, while the remaining four were allowed to air cool to room temperature. Longitudinal hardness surveys (Fig. 14) were made upon two ground flats spaced 180 degrees apart. Two 0.252-inch diameter by 1.0-inch gage length tensile specimens were machined and tested from each quenched and tempered double end

quench test bar for comparison with standard 0.505-inch diameter by 2.0-inch gage length specimens from the immersion quenched and tempered slices. The reproducibility of the mechanical properties is shown in Table II.

The variations from the mean in the as-quenched and as-tempered center hardness of the double end quench test bars (Fig. 14) appear to contradict the fine agreement indicated by the cooling data. Further examination of these and other specimens has shown, how-

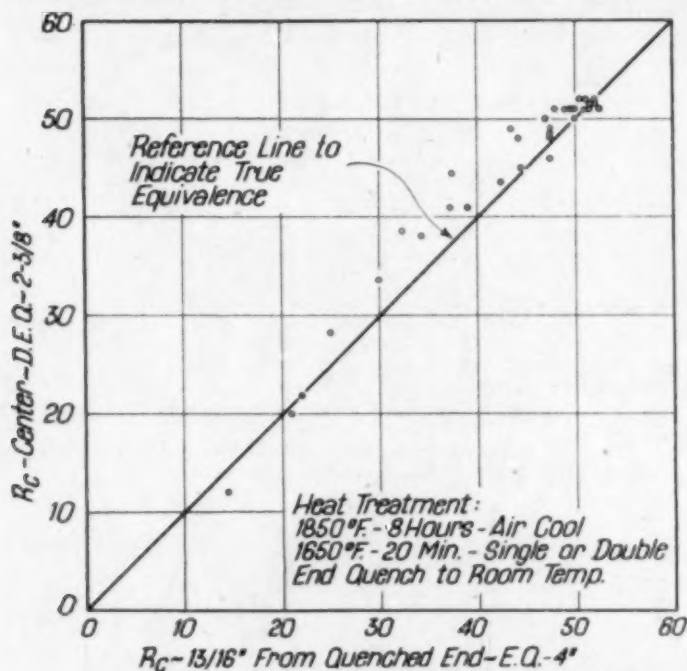


Fig. 15—Comparison of Rc Hardnesses at Positions on Single and Double End Quench Specimens Equivalent on the Basis of Cooling Rate at 1300 Degrees Fahr. Hardenability Data are from Thirty-Three Cast Alloy Steel Heats.

ever, that these variations resulted from heterogeneity of the cast material rather than from poor test reproducibility. The deviation in mechanical properties at the center of the double end quench test bars is no greater than that obtained for standard tensile bars.

PRACTICAL APPLICATION

The cooling rate at 1300 degrees Fahr. (705 degrees Cent.) is frequently employed as a criterion of quenching speed. The time required to cool from the temperature of quenching to some arbitrary level, such as 900 degrees Fahr. (480 degrees Cent.), also has served the same purpose. Correlation of these data for single end quench specimens (with technique standardized under A.S.T.M.

Designation A255-42T), and for various sections such as are employed industrially, has provided a basis for predicting the hardening characteristics of production parts. These criteria are quite useful for estimating the behavior of steels that transform above 900 to 1000 degrees Fahr. (480 to 540 degrees Cent.) as it can be shown that the cooling curves coincide closely above these temperatures.

If large amounts of alloying elements are present, as will be necessary for full hardening of heavy plates, prevention of the pearlitic transformation above 900 degrees Fahr. (480 degrees Cent.) is expected with any reasonably effective quench. Whereas simple steels that avoid the pearlitic reaction will usually transform to martensite, alloy steels may partially or wholly develop an acicular structure of the Bainite type because of the difference in shape of the respective "S" curves (Figs. 10 and 11). Thus for precise work, when acicular transformations are encountered, exact matching of cooling curves below, as well as above, 900 degrees Fahr. (480 degrees Cent.) may be desirable. The double end quench specimen and technique provide this matching for *plates*.

The hardnesses of comparable locations in the two different end quench specimens on the basis of cooling rate at 1300 degrees Fahr. (705 degrees Cent.) are compared in Fig. 15. The more rapid cooling of the double end quench test bar below 900 degrees Fahr. (480 degrees Cent.) is reflected in the somewhat higher hardnesses for those deep hardening steels that transform below this temperature. Practical experiments may establish that there is sufficient similarity for utility in many applications. However, certain factors indicate that caution is necessary. There is no assurance that similar hardness values mean similar structures. Mixtures (8) of different constituents may produce identical hardness but with quite different mechanical properties. Therefore, it is suggested that the double end quench specimen may be employed to predict behavior for the combination of heavy plate sections, of alloy steels that may not harden completely, and of critical requirements for certain mechanical properties in the center of the section.

Fig. 13 will allow selection of the double end quench specimen length that will closely match the center cooling of a given plate. A longitudinal hardness survey of the quenched specimen will then permit an excellent prediction of the cross sectional hardness of the plate, either as quenched or after tempering, without the necessity for

heat treating an inconveniently unwieldy plate or for assuming that a point on the single end quenched specimen is representative of the center of the plate at the same hardness level. After the hardness survey, the double end quench specimen may be machined to provide one or more tensile or impact specimens, and the mechanical properties measured directly. While this probably is not important for full hardening steels, it can be of vital concern in delineating the hardenability limitations if transformation begins in the range where separate ferrite rejection is possible.

While a separately cast bar may provide identical quenching rates and composition it does not insure heterogeneity similar to that of a heavy section with different solidification characteristics. As heterogeneity (9) is a factor influencing hardenability, this can become an important consideration in testing cast steels although it may be negligible for hot-worked alloys of similar composition. In many instances sound double end quench specimens machined from full size plates may be employed.

The efficiency of the cooling medium should also be considered. Variations in the severity of quench disturb the relationship of Fig. 13, which was developed for an agitated water bath at 95 degrees Fahr. With still water or oil, different double end quench specimen lengths would be required. Cooling of the quenched end of the hardenability specimen is so efficient that its rate of heat transfer probably exceeds the range in commercial practice. Compensation by lengthening the bar is thus a logical control measure.

If mechanical properties at the center of a plate are important and the double end quench specimen is employed to predict these, the probability that test pieces separately cast will furnish *maximum* properties, within grain size and heterogeneity limitations, should be noted. The cast bar is designed for soundness; the plate frequently contains at least microscopic shrinkage porosity. If properties reflecting all the variables in the actual plate are required, the double end quench specimens should be taken from it. This is seldom advisable, however, as the erratic results caused by either microscopic or macroscopic porosity are difficult to interpret: all investigations should be predicated upon sound test specimens.

SUMMARY AND CONCLUSIONS

1. Double end quench specimens and a hardenability testing technique have been developed and described.

2. Cooling curves for the comparison of single and double end quench specimens with lightly scaled plates quenched in agitated water have been obtained.

3. A useful correlation of specimen dimensions versus plate thickness, based on center cooling curves, has been presented.

4. Utility of the double end quench specimen for determining mechanical properties after typical plate heat treatment has been indicated.

5. Special applications where the double end quench technique may be superior to the single end quench practice have been suggested, and several variables discussed.

6. Future research might profitably extend the test bar length: plate thickness relation to heavier sections and delineate what allowances, if any, should be made for different quenching conditions.

7. Application of the 4:1 length: diameter ratio as employed for the double end quench bars might increase the utility of *single* end quenched specimens, which are currently limited to a useful length of 2 to 2¾ inches.

8. The problem of plate heterogeneity and its duplication in a separate test bar should also receive attention.

ACKNOWLEDGMENTS

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DISCUSSION

Written Discussion: By J. L. Waisman, metallurgist, and W. T. Snyder, assistant metallurgist, Douglas Aircraft Co., Inc., Chicago.

The authors are to be congratulated on developing their modification of the Jominy end quench test. They seem to have evolved an excellent means for accurate predictions on water-quenched plates.

Our work has shown us time and time again that the use of the standard end quench curve for making accurate predictions of the structure of oil-quenched bars is very limited for steels exhibiting a rapid intermediate transformation. This lack of accuracy is found for correlations based either on equivalent hardnesses or on equivalent cooling rates at 1300 degrees Fahr. (705 degrees Cent.). Accuracy in structural predictions with this type of steel (exhibiting a rapid intermediate transformation) would be expected only if the family of oil-quenched cooling curves were identical with those of the standard end quench test. Such, unfortunately, is hardly ever the case. The same limited structural correlations for some steels and quenching conditions which caused the writers to develop their new modification has also prompted us to use a somewhat similar modification. Although our test was sufficient for our needs, it was not as fully developed as the writers'. Two uses to which we put our test may suggest similar possibilities to the writers:

1. A considerable amount of interrupted quenching is done on heavy plates. The interruption of the quench may have two effects. First, the temperature gradient between surface and center will decrease. Secondly, an intermediate product may form in some steels which would not be found in any continuous cooling. An interruption of the standard end quench will cause a heat flow from hot to cold end. This heat flow may not be at all similar in magnitude to the heat flow from center to surface in an actual plate, and the standard end quench test therefore cannot be used to duplicate the conditions present in an interrupted quench. One of the chief advantages of our test was to duplicate interrupted quenching. The authors' test apparently can also be used to produce an accurate duplication of cooling curves during an interrupted quench. Has this been attempted?

2. We notice that, from Fig. 13, the length of the double end quench bar is somewhat greater in each case than the thickness of the equivalent plate. We wonder if it would not be possible to use a double end quench bar exactly equal to the thickness of the equivalent plate by changing the rate of water flow and/or by insulating the surfaces exposed to the air? If this were possible, specimens could be cut directly from plate thickness with their axes perpendicular to the plane of rolling (or plate surface) so that any segregation

present in the plate is in an equivalent location in the double end quench bar. A low hardenability core would be easily picked up by this method, whereas the present method of cutting the bar may miss it entirely. Since it is this center to surface segregation which is usual in rolled plates, such a modification may minimize the possibility of errors due to segregation mentioned by the authors.

Finally, do the authors think that oil could be used as the cooling medium in their test to more closely duplicate the oil quench family of cooling curves?

Authors' Reply

It is interesting that Messrs. Waisman and Snyder have encountered the same structural discrepancies between oil-quenched bars and the single end quench test that initiated the development of the double end quench correlation for flat plates.

Cooling curves have not been obtained for interrupted quenching techniques, but the test bar or plate may be time quenched to any desired temperature. Plant practices for alloy steel plates involved time quenching to 400 to 500 degrees Fahr. (205 to 260 degrees Cent.) in still water. The less severe quench is evident from a comparison of laboratory and plant practices:

Quenching Time for Casting to Reach 400 to 500 degrees Fahr. (Austenitized 1650 degrees Fahr.)	
2-inch Plate (Plant—Still Water)	160 seconds
1½-inch Plate (Laboratory—Agitated Water)	118 seconds
D.E.Q.—2.15-inch Test Bar	113 seconds

The need for test bars longer than the equivalent plate thicknesses for the smaller sizes introduces a difficulty in employing specimens cut from transverse sections of actual plates that has not yet been surmounted. Studies involving the use of composite bars, which have short sections welded to the ends, are in progress. If satisfactory heat flow across the weld junction can be attained it is anticipated that bars cut transversely from a plate may be extended in length to provide the correlating specimen without sacrificing the cast plate center structure.

Little variation in quenching rate with changing water flow has been discovered within usual limits; it is controlled but its modification has not been considered of value or importance. Substitution of oil for water appears practical if the liquid is employed in a closed system. Cooling curves for this technique are not available, however.

Insulation of the air-cooled cylindrical face will retard the cooling of the specimen but the quantitative effect has not been delineated.

ISOTHERMAL TRANSFORMATION AND END-QUENCH HARDENABILITY OF SOME NE STEELS

BY R. L. RICKETT, J. G. CUTTON, C. B. BERNHART, JR.,
AND J. R. MILLIKIN

Abstract

The isothermal transformation diagram was determined for each of the following NE steels: 8620, 8744 (8642), 8949, 9420, 9442, 9540, 9642 and 9650; and for comparison the end-quench hardenability of these or similar steels was measured.

These isothermal transformation diagrams are of the general type previously found for many S.A.E. alloy steels. All exhibit a bulge, to the right, in the end-of-transformation line, usually in the neighborhood of 1000 degrees Fahr. (540 degrees Cent.). In this same temperature range there is a "shelf" or "secondary nose" in the curve which represents the beginning of ferrite-carbide aggregate formation. The presence of this "shelf" means that it is possible to obtain intermediate-temperature transformation products in such steels on continuous cooling, provided the cooling rate is within a particular range.

The microstructure of these NE steels when isothermally transformed likewise resembles that of some S.A.E. alloy steels. At temperatures above about 1000 degrees Fahr. (540 degrees Cent.) proeutectoid ferrite and pearlite are formed. Around 900 to 1000 degrees Fahr. acicular ferrite containing "dots", presumably carbide particles, forms; in some of the steels this is followed by formation of a dense, dark-etching constituent during the later stages of transformation. At still lower temperatures an acicular iron-carbide aggregate (bainite) is formed. The appearance of these transformation products, particularly those formed at 1000 degrees Fahr. (540 degrees Cent.) and below, varies with the carbon content of the steel.

The end-quench curves indicate the relative hardenability that would be predicted from the isothermal transformation diagrams; NE 8949 and NE 9540 are particu-

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larly deep hardening steels. The curve for NE 9650 exhibits a "hump" associated with a change in type of transformation product which can be explained on the basis of the isothermal transformation diagram for this steel. The isothermal transformation diagram furnishes much more information than the end-quench curve, but the latter is a more precise measure of hardenability; to a considerable extent the isothermal transformation diagram and end-quench test supplement each other.

INTRODUCTION

THE isothermal transformation diagram is now being used extensively in connection with practical heat treating, particularly in normalizing or annealing treatments employed to produce a definite hardness or microstructure desired for some subsequent operation, such as machining. As an example, at one steel mill it has been found that, for alloy steel, the use of annealing schedules based on isothermal transformation data resulted in a substantial saving in time and expense as compared to the slow cooling cycles formerly used. When the NE (National Emergency) steels were first proposed, little was known concerning their isothermal transformation behavior and many requests for such data were received. Consequently, we determined the diagram for eight representative steels, including two carburizing grades, NE 8620 and NE 9420, and several in the 0.40 to 0.50 per cent carbon range. A few of these diagrams have been issued previously;¹ others are given here for the first time.

In addition to isothermal transformation diagrams, the end-quench hardenability curve for several of the steels is also presented. In some instances, however, the stock used in the isothermal transformation work was not large enough in cross section to permit making standard end-quench specimens; in these instances other steels of similar composition were used. These end-quench curves are included for comparison with the isothermal diagrams; also, they may be useful to those accustomed to evaluate the heat treating characteristics of a steel from its end-quench hardenability curve.

Changes made in the NE steels from time to time as the availability of alloying elements varied or experience indicated such changes to be desirable have made some of the steels obsolete, in a

¹"Atlas of Isothermal Transformation Diagrams," U. S. Steel Corporation of Delaware, Pittsburgh, 1943.

sense, even before their properties and degree of usefulness were fully established. Some of the steels included in the present investigation are in this category, but the information given concerning them may still be found useful since some of them do not differ too greatly from steels that are now being used. Moreover, there is now available only a limited amount of data regarding the effect of specific combinations of alloying elements on isothermal transformation behavior, and further information of this sort should lead to a better understanding of the functions of alloying elements in steel.

STEELS INVESTIGATED

Table I presents the composition of the steels used; these were selected to represent the principal NE types available at the time the work was done. Steels NE 8744, NE 8949, NE 9642 and NE 9650

Table I
Composition of Steels Used

S-Curve or Steel Hardenability*		Composition—Per Cent (Check Analysis)								
		C	Mn	P	S	Si	Cr	Ni	Mo	Zr
NE8620	Both	0.18	0.79	0.021	0.023	0.31	0.56	0.52	0.19
NE8744	Both	0.44	0.90	0.019	0.031	0.25	0.54	0.44	0.22
NE8949	Both	0.52	1.19	0.021	0.028	0.28	0.51	0.53	0.35
NE9420	Both	0.24	0.94	0.020	0.032	0.47	0.34	0.30	0.14
NE9435	E-Q	0.38	1.06	0.016	0.029	0.55	0.35	0.31	0.15
NE9442	S-Curve	0.38	1.08	0.70	0.40	0.34	0.11	0.030
NE9540	S-Curve	0.38	1.45	0.66	0.52	0.57	0.19	0.015
NE9542	E-Q	0.44	1.36	0.018	0.036	0.53	0.53	0.49	0.24
NE9642	S-Curve	0.39	1.44	0.56	0.53	0.06	0.01	0.020
NE9650	S-Curve	0.48	1.30	0.59	0.54	0.06	0.01	0.025
NE9650	E-Q	0.51	1.38	0.019	0.039	0.51	0.53	0.04	0.03

*End-Quench Hardenability (E-Q).

have since been discontinued. The NE 9420, NE 9435, NE 9442, NE 9540 and NE 9542 steels used in this investigation are higher in silicon than the range 0.20 to 0.35 per cent now specified. This NE 9420 steel is also higher in carbon than specified for that grade but within the range of NE 9422; the NE 9442 used is low in carbon and is more properly an NE 9437 or NE 9440. It should be noted that the NE 9442, NE 9540, NE 9642 and NE 9650 steels used in the isothermal transformation work contain zirconium, added, presumably, as a deoxidizer. The effect this zirconium may have had on the isothermal transformation behavior is not known with certainty but is believed to be slight.

In Table II are listed NE steels currently made that are similar

Table II
Current NE Steels* Whose Composition Includes or Approximates That of Steels
for Which S-Curves Are Given

Steel	Composition	Composition—Per Cent					
		C	Mn	Si	Cr	Ni	Mo
NE8620	Actual	0.18	0.79	0.31	0.56	0.52	0.19
NE8620	Specified	0.18-0.23	0.70-0.90	0.20-0.35	0.40-0.60	0.40-0.70	0.15-0.25
NE8720	Specified	0.18-0.23	0.70-0.90	0.20-0.35	0.40-0.60	0.40-0.70	0.20-0.36
NE9420	Actual	0.24	0.94	0.47	0.34	0.30	0.14
NE9420	Specified	0.18-0.23	0.80-1.10	0.20-0.35	0.30-0.50	0.30-0.60	0.08-0.15
NE9422	Specified	0.20-0.25	0.80-1.10	0.20-0.35	0.30-0.50	0.30-0.60	0.08-0.15
NE9425	Specified	0.23-0.28	0.80-1.10	0.20-0.35	0.30-0.50	0.30-0.60	0.08-0.15
NE8744	Actual	0.44	0.90	0.25	0.54	0.44	0.22
NE8642	Specified	0.40-0.45	0.75-1.00	0.20-0.35	0.40-0.60	0.40-0.70	0.15-0.25
NE8645	Specified	0.43-0.48	0.75-1.00	0.20-0.35	0.40-0.60	0.40-0.70	0.15-0.25
NE8949	Actual	0.52	1.19	0.28	0.51	0.53	0.35
NE9550	Specified	0.48-0.53	1.20-1.50	0.40-0.60	0.40-0.60	0.40-0.70	0.15-0.25
NE9442	Actual	0.38	1.08	0.70	0.40	0.34	0.11
NE9442	Specified	0.40-0.45	1.00-1.30	0.20-0.35	0.30-0.50	0.30-0.60	0.08-0.15
NE9437	Specified	0.35-0.40	0.90-1.20	0.20-0.35	0.30-0.50	0.30-0.60	0.08-0.15
NE9440	Specified	0.38-0.43	0.90-1.20	0.20-0.35	0.30-0.50	0.30-0.60	0.08-0.15
NE9540	Actual	0.38	1.45	0.66	0.52	0.57	0.19
NE9540	Specified	0.38-0.43	1.20-1.50	0.40-0.60	0.40-0.60	0.40-0.70	0.15-0.25
NE9537	Specified	0.35-0.40	1.20-1.50	0.40-0.60	0.40-0.60	0.40-0.70	0.15-0.25

*Specified compositions are as given in American Iron and Steel Institute Revised List of August 15, 1943.

in composition to those for which isothermal transformation diagrams are given. The diagrams presented should be useful as a guide in the heat treatment of these "similar" grades of steel.

PROCEDURE

These isothermal transformation diagrams were determined by the metallographic method, which has been described elsewhere.^{2, 3} Transformation was considered to have started when definite evidence of its occurrence could be detected in at least one out of every 3 or 4 fields examined with the microscope. In many instances the length of time required to produce this much transformation was considerably less than for 1 per cent transformation, the criterion adopted by some other investigators.

Another factor entering into the determination and interpretation of the isothermal transformation diagram is segregation, which is of two types: (a) "microscopic" segregation, which leads to banding; (b) "macroscopic" segregation which results in a gradual change in composition from surface to center or top to bottom of the ingot. Either type may lead to rather marked differences in rate

²E. S. Davenport and E. C. Bain, "Transformation of Austenite at Constant Subcritical Temperatures," *Transactions*, American Institute of Mining and Metallurgical Engineers, Iron and Steel Division, Vol. 90, 1930, p. 117-154.

³E. S. Davenport, "Isothermal Transformation in Steels," *TRANSACTIONS*, American Society for Metals, Vol. 27, 1939, p. 837-886.

of transformation between portions of different composition. These specimens previously had been normalized but nothing further was done to minimize or eliminate segregation. So far as banding is concerned, in the diagrams presented here the "start of transformation" line is based on the first bands to transform and the "end of transformation" line on the last bands to transform. An attempt was made to minimize the effect of over-all segregation (macroscopic) by considering only that portion of the specimen representing the location midway between surface and center of the original bar. It should be recognized that if marked over-all segregation is present the rate of transformation in various parts of a bar may vary considerably from that in this midway location.

The Ae_1 and Ae_3 temperatures shown on the isothermal transformation diagrams were determined to within ± 5 degrees Fahr. by heating normalized specimens in molten lead at a series of selected temperatures. These specimens were held at temperature for 1 to 4 hours, then quenched and examined metallographically; their hardness was also measured. In alloy steels, austenite, ferrite and carbide may exist in equilibrium over a temperature range; the Ae_1 temperature shown in the diagram is the lower limit of this range, the extent of which was not determined.

The temperature at which martensite starts to form on quenching, the so-called M_s point, was not determined experimentally for any of these steels. An attempt was made to do so for NE 8620 and NE 9420 but was unsuccessful because of the rapid rate of isothermal transformation at temperatures in the neighborhood of the expected M_s point and at the higher temperature employed to temper any martensite that might have formed. The approximate M_s point for each of the higher-carbon steels, calculated from an equation* similar to that published by Payson and Savage,⁴ is indicated roughly by the location of the lower end of the start-of-transformation line in the diagram for each of these steels.

End-quench hardenability was determined in accordance with

⁴P. Payson and C. H. Savage, "Martensite Reactions in Alloy Steels," *TRANSACTIONS, American Society for Metals*, Vol. 33, 1944, p. 261-275.

*The equation used is $M_s(^{\circ}F) = 965 - 570C - 100Mn - 30Ni - 27Cr - 110Mo$, where C, Mn, etc., are the amount, in per cent, of these elements present in the steel. This equation, devised by T. W. Merrill and R. A. Grange of the U. S. Steel Corporation Research Laboratory in 1940, is based on published data available at that time. For the particular steels to which it was applied, this equation gives values of the M_s temperature which differ by only 2-25 degrees Fahr. from those calculated by means of the Payson and Savage equation. While later information indicates that this equation should be modified to be generally applicable, it has been shown experimentally to give fairly reliable results when applied to medium-carbon low-alloy steels, as in this instance.

A.S.T.M. Specification A255-42T. Both regular (1-inch diameter) and Type L specimens of NE 8620 and NE 9420 were tested and regular specimens of the other steels. To protect them from scaling and decarburization they were heated in containers only slightly larger in diameter than the specimens and were covered with powdered charcoal. Total time in the furnace was 1.5 hours, of which nearly 1 hour was required to bring the specimen up to temperature.

RESULTS

Carburizing Grades, NE 8620 and NE 9420—The isothermal transformation diagrams for these two steels, Figs. 1 and 2, are quite similar. It should be noted that the time scale in these diagrams ex-

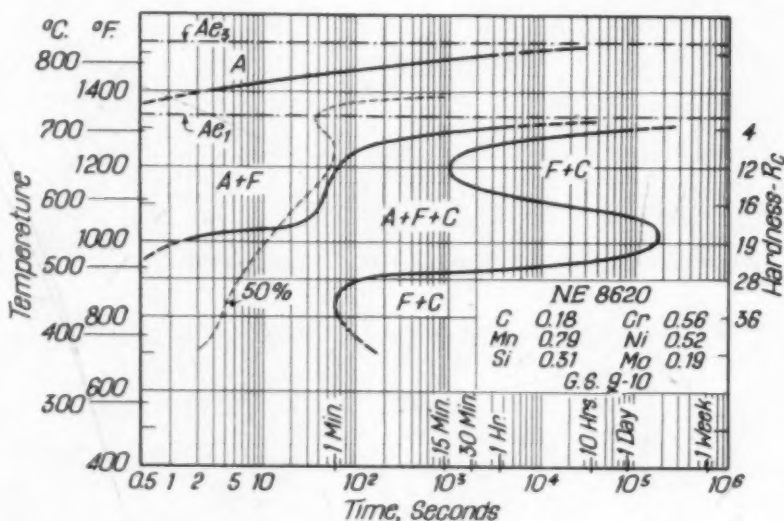


Fig. 1—Isothermal Transformation Diagram for NE 8620 (Approx. NE 8720) Austenitized at 1650 Degrees Fahr.

tends to 1,000,000 seconds which is a longer period than that covered by the other diagrams. In both steels, transformation begins in less than 1 to 2 seconds at 1400 degrees Fahr. (760 degrees Cent.) or below. At temperatures above about 1000 degrees Fahr. (540 degrees Cent.) the first product formed is ferrite, whereas at lower temperatures the initial transformation product is an aggregate of ferrite and carbide. In common with the diagrams for other steels that contain chromium or molybdenum, there is a bulge, to the right, in the end-of-transformation line; this bulge is more pronounced

and at a slightly lower temperature in NE 8620 than in NE 9420. In the temperature range 1200 to 1050 degrees Fahr. (650 to 565 degrees Cent.), formation of ferrite-carbide aggregate starts somewhat sooner in NE 9420 than in NE 8620. For both steels at temperatures above 1000 degrees Fahr. (540 degrees Cent.), transformation is completed within less time at 1200 degrees Fahr. (650 degrees Cent.) than at any other temperature; this, therefore, is the

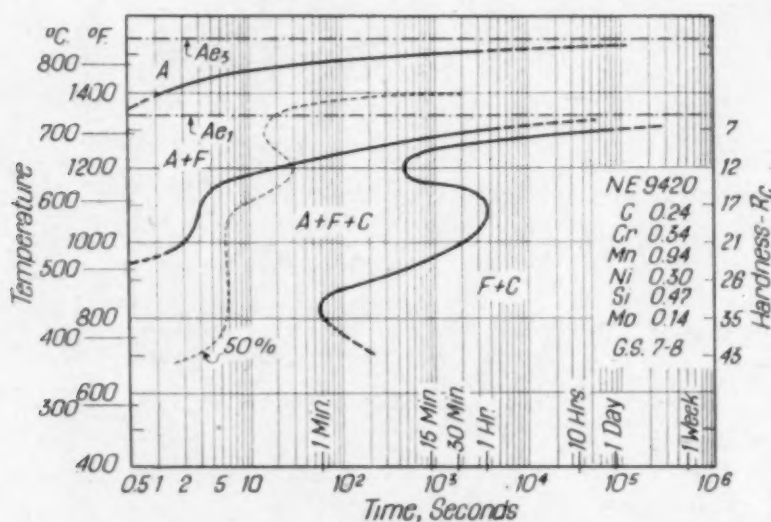


Fig. 2—Isothermal Transformation Diagram for NE 9420 (NE 9422-9425) Austenitized at 1650 Degrees Fahr.

temperature at which these steels can be isothermally annealed in the shortest period of time.

Microstructures of NE 8620 transformed isothermally at various temperatures are shown in Figs. 3 and 4; the structures of NE 9420 are so similar that no photomicrographs were made. The products of transformation at 1300 to 1000 degrees Fahr. (705 to 540 degrees Cent.) (Fig. 3) are ferrite, pearlite, and isolated carbide particles. The pearlite formed is less distinctly lamellar and less easily resolved (finer) the lower the temperature at which transformation occurs; on transformation at 1000 degrees Fahr. (540 degrees Cent.) it tends to form a network pattern. A marked difference in microstructure may be noted between the 1300 and 1200-degree Fahr. (705 and 650-degree Cent.) transformation products, Figs. 3A and 3B. Transformation products formed at 900 degrees Fahr. (480 degrees Cent.) and below are somewhat acicular, although after complete transformation at 800 degrees Fahr. (425 degrees Cent.) (Fig. 4D) or 900 degrees Fahr. (480 degrees Cent.)

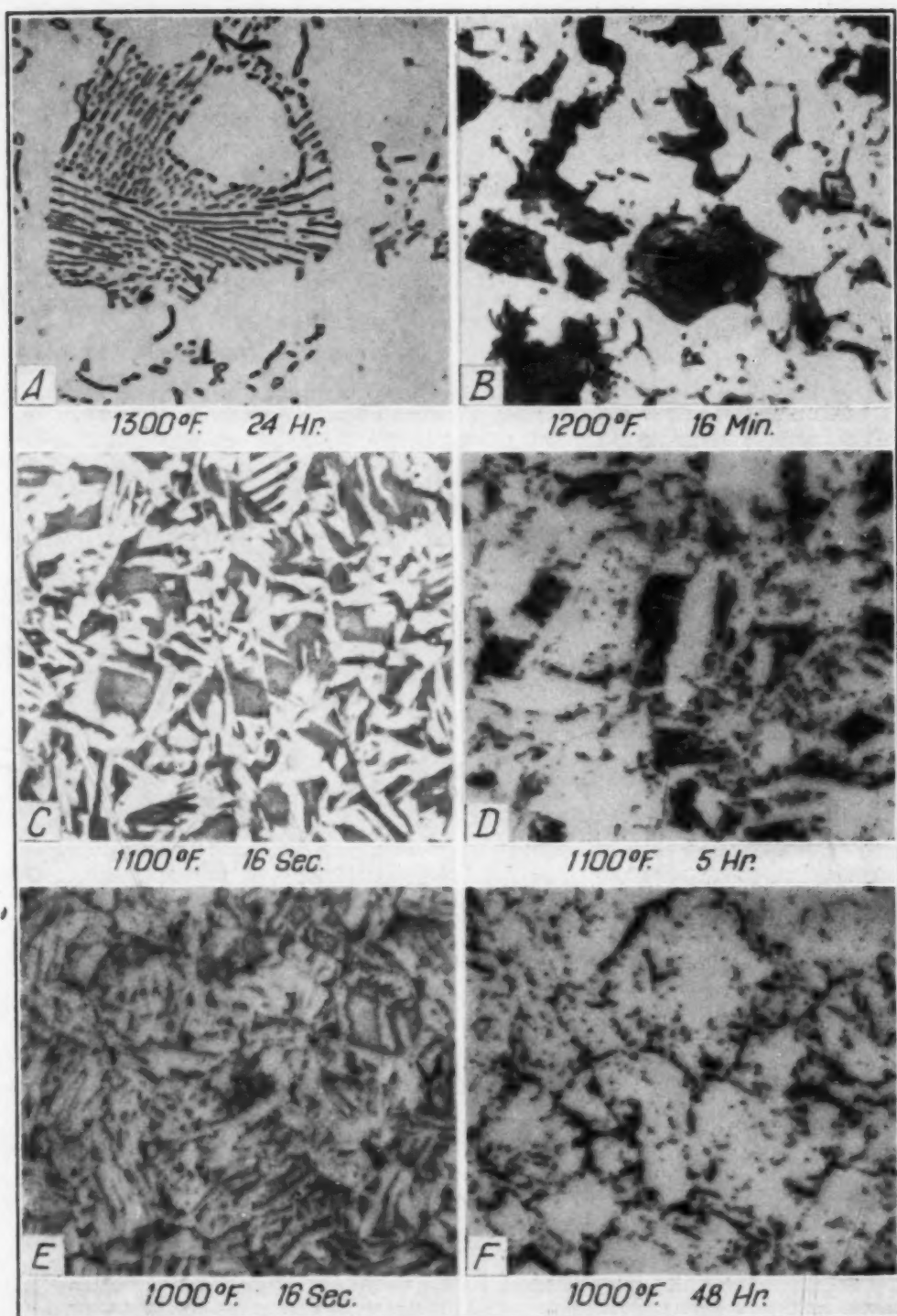


Fig. 3—Microstructure of NE 8620 Isothermally Transformed at 1300 to 1000 Degrees Fahr. Picral Etch. $\times 1000$.

(Fig. 4B) this acicular tendency may be only faintly evident. When partially transformed at 700 degrees Fahr. (370 degrees Cent.), Fig. 4E, the microstructure is somewhat different than after trans-

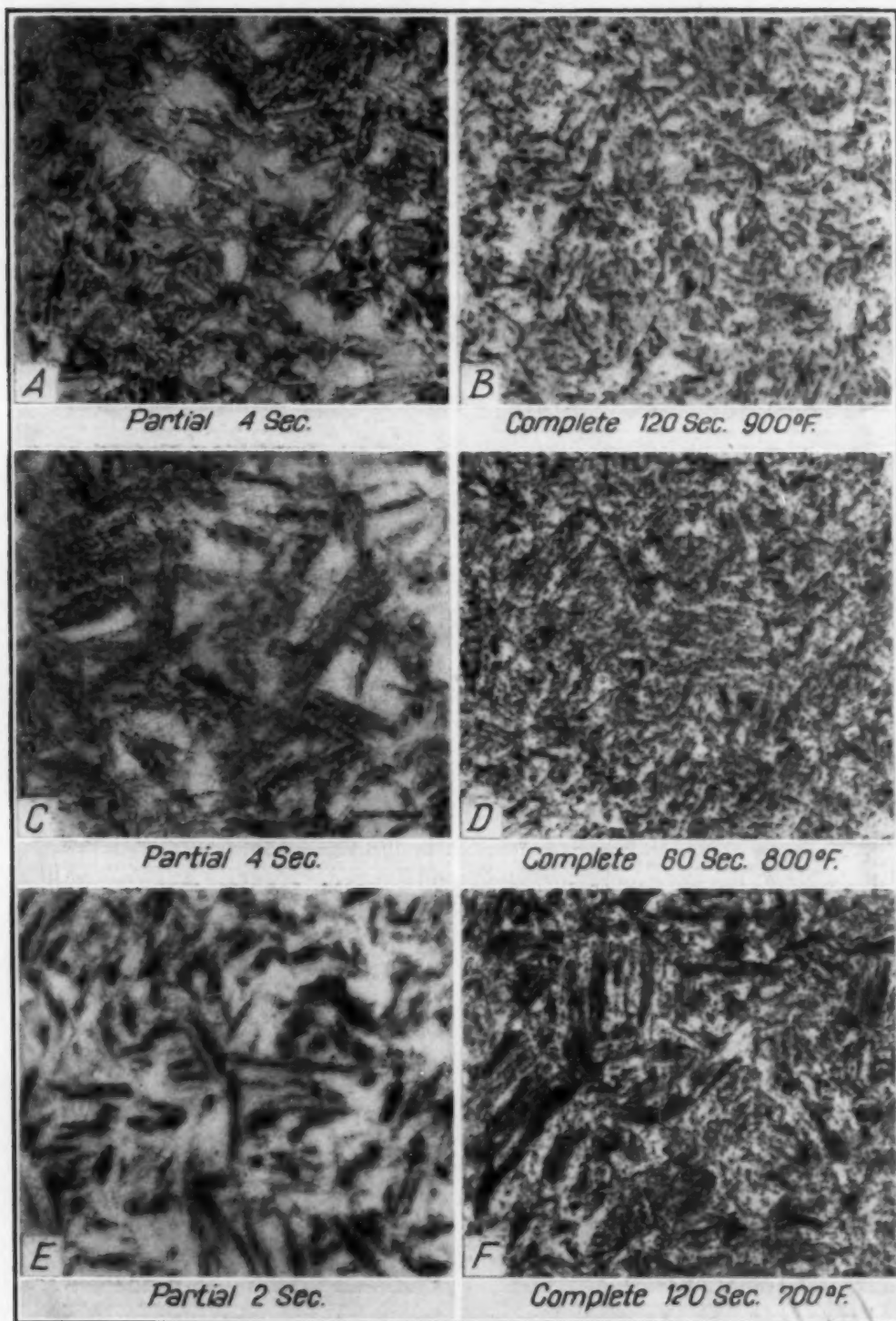


Fig. 4—Microstructure of NE 8620 Partially or Completely Transformed Isothermally at 900 to 700 Degrees Fahr. Picral Etch. $\times 1000$.

formation at higher temperatures. In Fig. 4E may be seen a blunt acicular product; this may be martensite formed when the specimen cooled to 700 degrees Fahr. (370 degrees Cent.), and subsequently

tempered at that temperature. After complete transformation, Fig. 4F, the microstructure consists of these blunt needles and another product which probably formed isothermally and is similar to that formed at 800 degrees Fahr. (425 degrees Cent.), Fig. 4D.

End-quench hardenability curves for these two steels, Fig. 5A, are very similar; hardness at the quenched end is slightly higher in NE 9420, because of its higher carbon content, than in NE 8620. Both steels are shallow hardening because of the low carbon content.

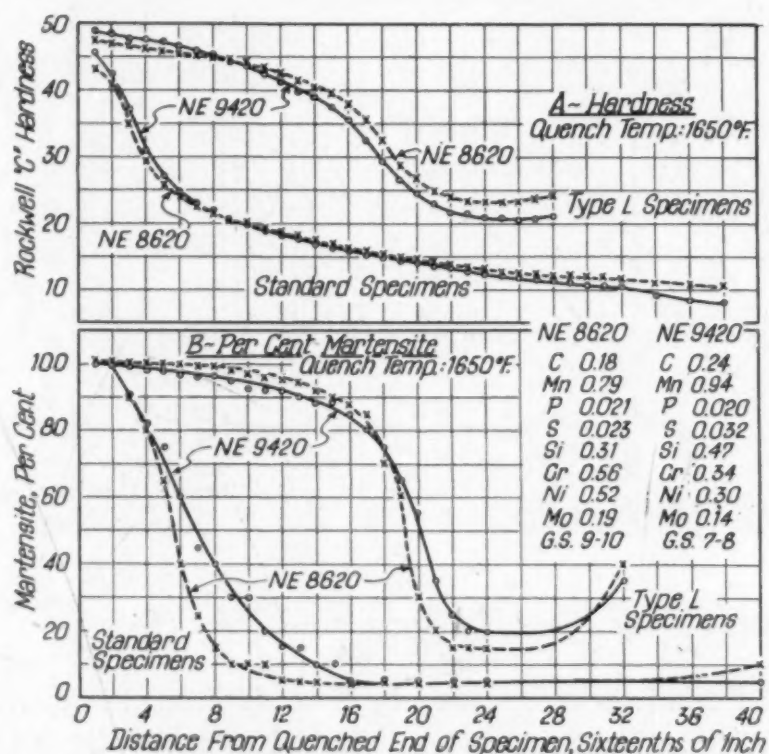


Fig. 5—End-Quench Hardenability of NE 8620 and NE 9420.

However, as shown in Fig. 5B which is based on the results of metallographic examination, martensite is present even near the slowly cooled end of specimens of each steel. This martensite is greater in amount in the more slowly transforming bands but is by no means confined to them. The curves shown in Fig. 5B also indicate that the relative hardenability of the two steels varies according to the degree of transformation for which the comparison is made.

Medium-carbon Mn-Cr-Ni-Mo Steels—Isothermal transformation diagrams for four steels of this type, NE 8744, NE 8949, NE 9442 and NE 9540, are given in Figs. 6 to 9. These diagrams, which

extend to 100,000 seconds, exhibit several features in common. One of these features is the bulge, to the right, in the end-of-transformation line, which is also present in the NE 8620 and NE 9420 diagrams (Figs. 1 and 2); another is the "shelf", at approximately 1000 degrees Fahr. (540 degrees Cent.), in the line which represents the start of carbide formation. A third feature of all the diagrams, except that for NE 8744 (Fig. 6), is a change in type of transforma-

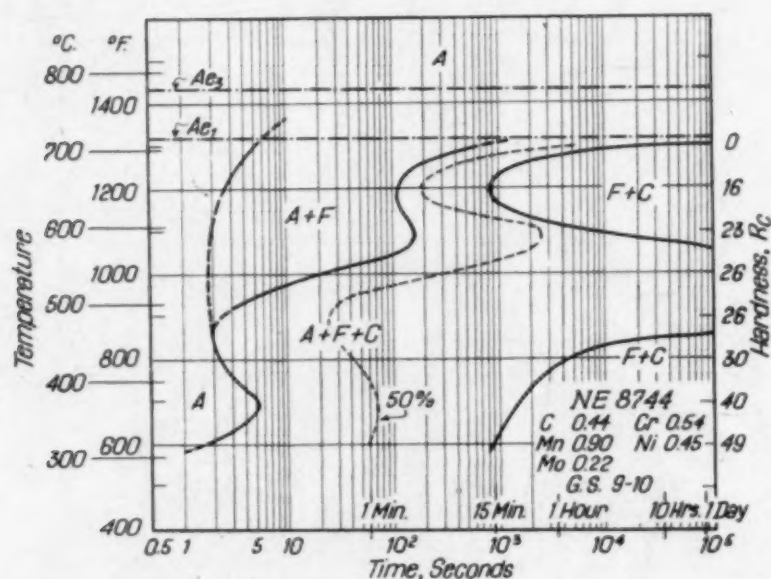


Fig. 6—Isothermal Transformation Diagram for NE 8744 (NE 8642-8645) Austenitized at 1550 Degrees Fahr.

tion product which is represented by a heavy dashed line within the $A+F+C$ field. This change is discussed in more detail below in connection with the diagram for NE 9650. This line is not shown in the diagram for NE 8744 (Fig. 6), though that steel also undergoes a similar change in type of transformation product, because the exact location of the line was not established satisfactorily. For all four steels in the temperature range above 1000 degrees Fahr. (540 degrees Cent.), transformation is most rapid at about 1200 degrees Fahr. (650 degrees Cent.) and hardness after complete transformation at this temperature differs little among them.

Although for NE 8744 (Fig. 6) the pearlite "nose" is at about 100 seconds, ferrite begins to form in a much shorter time. A fairly fast cooling rate is required, therefore, if formation of high-temperature transformation products is to be avoided completely. Since this steel is within the NE 8642-8645 composition range, it is of interest

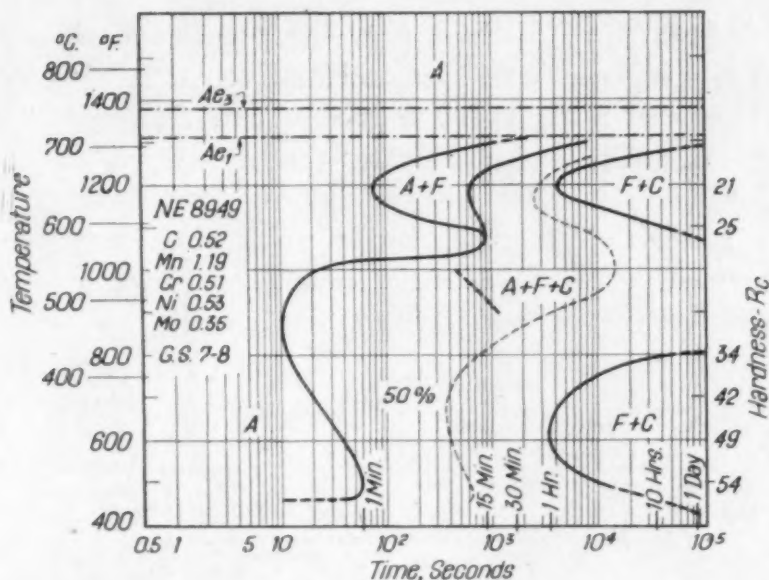


Fig. 7—Isothermal Transformation Diagram for NE 8949 (Approx. NE 9550) Austenitized at 1550 Degrees Fahr.

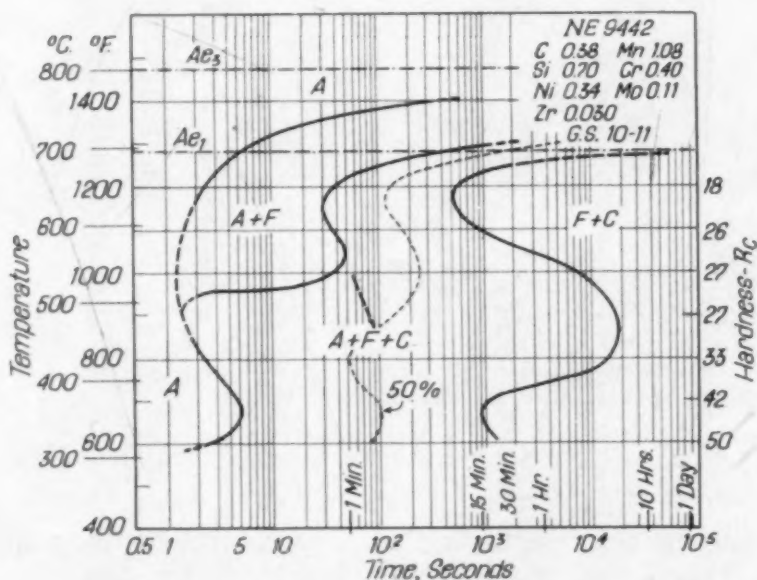


Fig. 8—Isothermal Transformation Diagram for NE 9442 (NE 9437-9440) Austenitized at 1575 Degrees Fahr.

to compare Fig. 6 with Fig. 1, the diagram for NE 8620, keeping in mind that in Fig. 1 the time scale extends to 1,000,000 seconds, but in Fig. 6 to only 100,000 seconds. These two diagrams show that the effect of lower carbon on transformation above 1000 degrees Fahr. (540 degrees Cent.) is to move the beginning line to the left without greatly affecting the ending line; below 1000 degrees Fahr. (540 degrees Cent.) the ending line is also moved to the left.

The diagram for NE 8949, Fig. 7, is consistent with the known deep-hardening character of this steel. Even the lower "nose", at about 900 degrees Fahr. (480 degrees Cent.), is quite far to the right in the diagram, which indicates that fairly large sections may be quenched without obtaining any intermediate-temperature transformation product. The suppression of ferrite formation and of transformation in the intermediate temperature range is partially due to the relatively high carbon content of this steel.

NE 9442, Fig. 8, is similar to NE 8744 (Fig. 6) in its isothermal transformation behavior, except that it transforms a little more

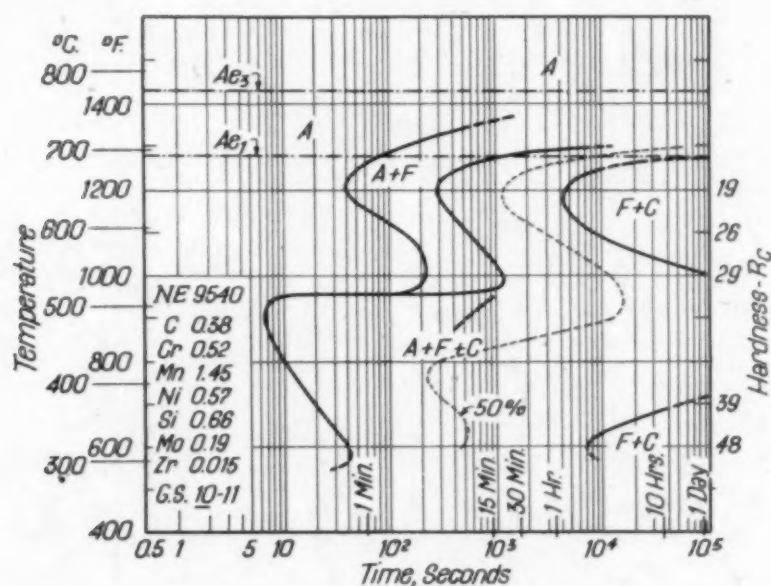


Fig. 9—Isothermal Transformation Diagram for NE 9540 (NE 9537) Austenitized at 1525 Degrees Fahr.

rapidly, particularly at temperatures around 900 to 1200 degrees Fahr. (480 to 650 degrees Cent.).

The diagram for NE 9540, Fig. 9, is somewhat like that for NE 8949, Fig. 7, as would be expected since these two steels are of the same general type. A comparison of the diagram for NE 9540, Fig. 9, with that for NE 8744, Fig. 6, illustrates the effect of manganese; in the former (1.45 per cent manganese) the ferrite line, the beginning-of-pearlite line, and the 900-degree Fahr. (480-degree Cent.) "nose" of the diagram are considerably to the right of the corresponding parts of the diagram for NE 8744 (0.90 per cent manganese).

The microstructures of these steels when isothermally transformed are, in general, similar to those of S.A.E. 4140, which have

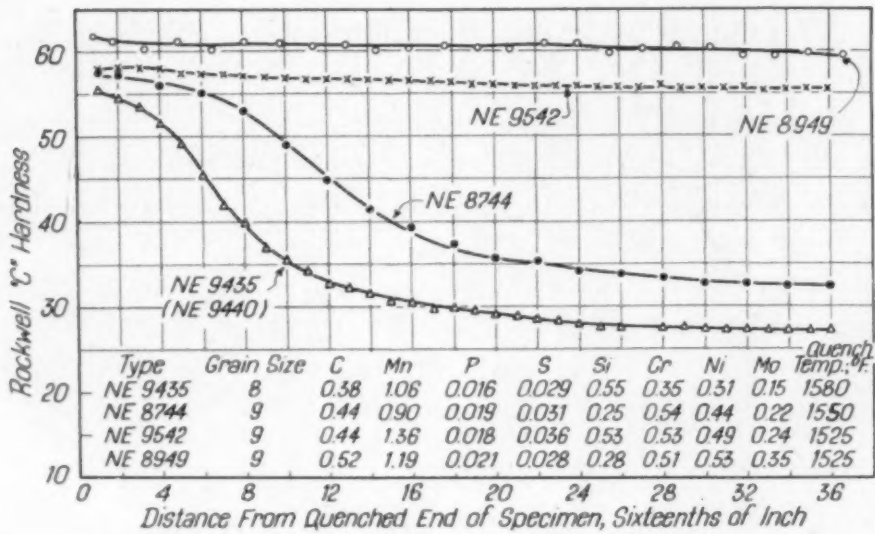


Fig. 10—End-Quench Hardenability of Four Manganese-Chromium-Nickel-Molybdenum NE Steels.

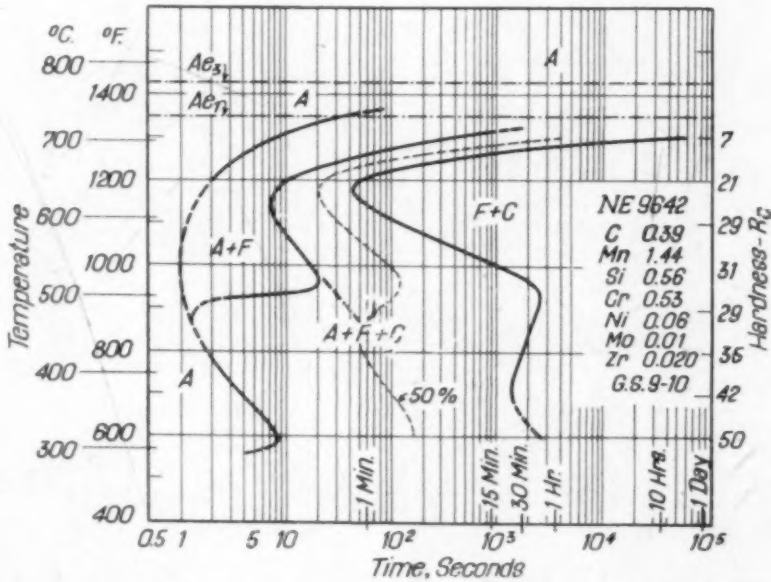


Fig. 11—Isothermal Transformation Diagram for NE 9642 Austenitized at 1525 Degrees Fahr.

been described in the literature.^{3, 5} They also resemble to some extent those found in NE 9650 which are described below.

End-quench hardenability curves for these same steels or similar ones are shown in Fig. 10. The relative hardenability of the four steels is the same as would be expected from the isothermal transformation diagrams. The end-quenched specimens of NE 8949 and

⁵E. S. Davenport, "Transformation in S.A.E. 4140," METAL PROGRESS, Vol. 38, 1940, p. 171, 177.

NE 9542 hardened throughout practically their whole length. The other two steels, NE 9435 (similar in composition to NE 9442) and NE 8744 are not as deep hardening; metallographic examination of the specimens revealed, however, that some martensite is present in each as far as $2\frac{1}{2}$ inches from the quenched end and that this martensite is not confined solely to the more slowly transforming bands.

Medium-carbon Mn-Cr-Si Steels—Two steels of this type, NE 9642 and NE 9650, were investigated with results presented in Figs.

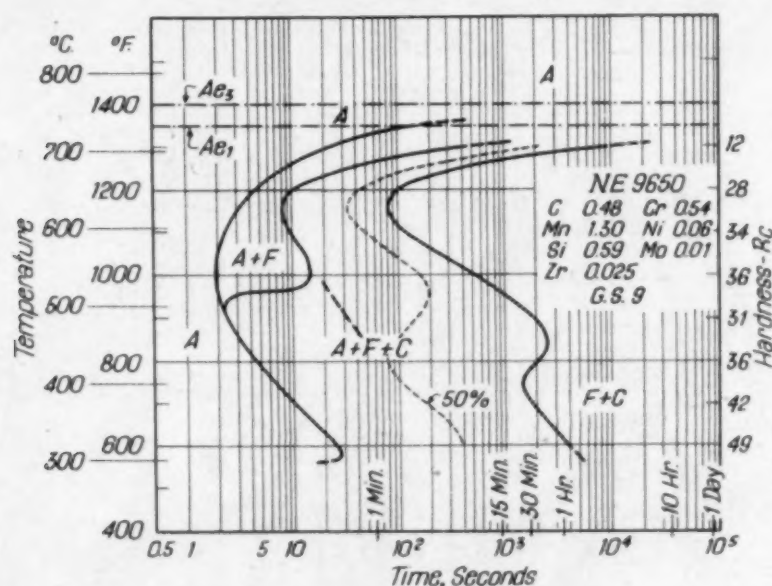


Fig. 12—Isothermal Transformation Diagram for NE 9650 Austenitized at 1525 Degrees Fahr.

11 and 12 which are very similar, as would be expected, since the steels differ only slightly in composition, mainly in carbon and manganese content. In the lower carbon steel, transformation begins slightly sooner than in the higher carbon steel but there is little difference between them in the time required for complete transformation. For both steels, the hardness after complete transformation increases in normal fashion as the transformation temperature decreases, except between 1000 and 800 degrees Fahr. (540 and 425 degrees Cent.), in which range there is an anomalous drop in hardness which has been noted in other steels and which is associated with the marked change in microstructure described below.

In Fig. 13 are shown the microstructures of NE 9650 austenitized at 2000 degrees Fahr. (1095 degrees Cent.), then isothermally transformed at various temperatures. The purpose of using this relatively high austenitizing temperature was to coarsen the austenite

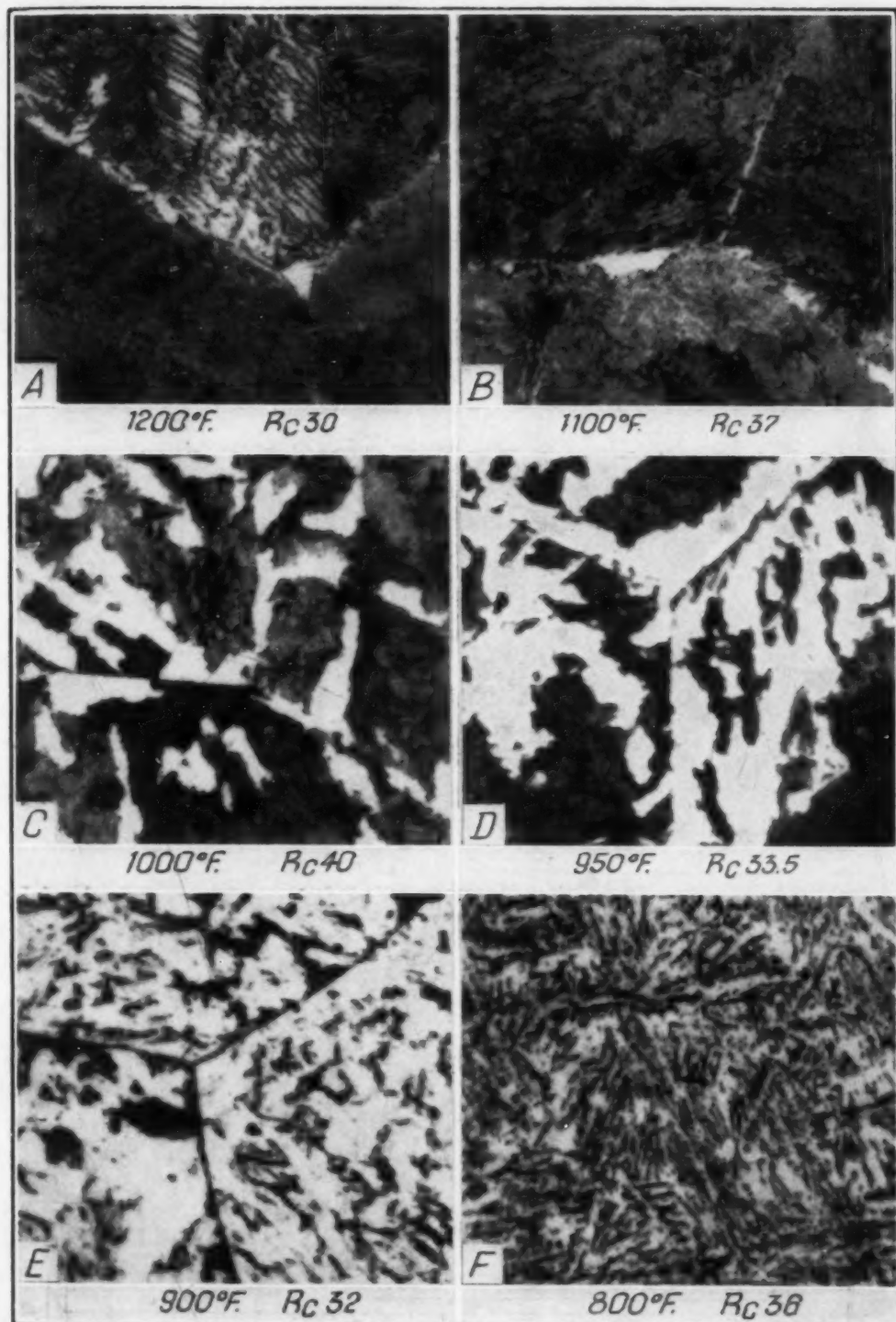


Fig. 13—Microstructure of NE 9650 Austenitized at 2000 Degrees Fahr. and Completely Transformed Isothermally at 1200 to 800 Degrees Fahr. Picral Etch. $\times 2000$.

grains (average grain size 5) and thus render the course of subsequent isothermal transformation somewhat easier to follow metal-



Fig. 14—Change in Type of Isothermal Transformation Product at 900 Degrees Fahr. in NE 9650 Austenitized at 2000 Degrees Fahr. Picral Etch. $\times 2000$.

lographically. In the temperature range from 1300 degrees Fahr. (705 degrees Cent.) down to 1100 degrees Fahr. (595 degrees

Cent.), pearlite and a small amount of proeutectoid ferrite are formed, the pearlite being finer and the amount of proeutectoid ferrite being less the lower the transformation temperature. Transformation at 1000 to 900 degrees Fahr. (540 to 480 degrees Cent.), however, yields an increased amount of ferrite (see Figs. 13C-13E) which is evidently responsible for the drop in hardness in this range. To some extent these microstructures are representative of the other

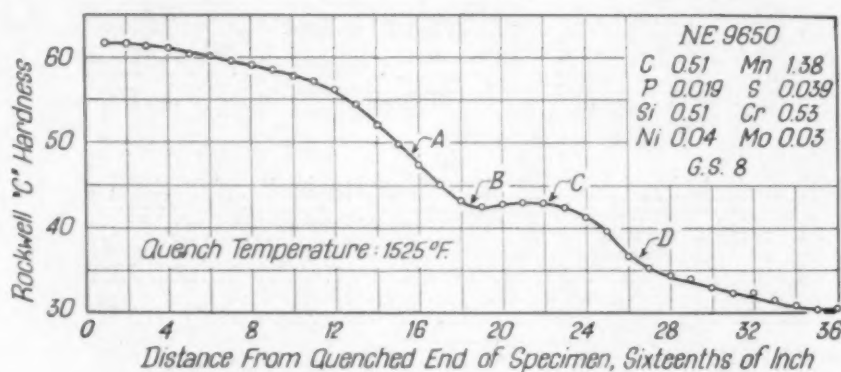


Fig. 15—End-Quench Hardenability of NE 9650.

medium-carbon NE steels investigated. In those of lower carbon content, however, more ferrite is formed at temperatures above 1000 degrees Fahr. (540 degrees Cent.) than in NE 9650.

In Figs. 11 and 12, as in the diagrams for most of the other medium-carbon steels, the heavy dashed line drawn within the A+F+C field in the 1000 to 900-degree Fahr. (540 to 480-degree Cent.) region represents a change in the type of transformation products which has been discussed previously in the literature.^{3, 6} This change for NE 9650 at the 900-degree Fahr. (480-degree Cent.) level is illustrated in Fig. 14. The first product to form, Fig. 14A, is acicular ferrite within which are black dots, presumably carbide particles, a structure sometimes referred to as the "X" constituent. As transformation proceeds, the amount of this product increases and the concentration of black dots within it becomes greater, Fig. 14B. Eventually, as shown in Fig. 14C, there appears a different type of product which occurs as a partial network in prior austenite grain boundaries and as a "chunky" dark-etching constituent at the boundaries and within prior austenite grains.

The end-quench hardenability curve of NE 9650, shown in Fig.

⁶E. S. Davenport, R. A. Grange and R. J. Hafsten, "Influence of Austenite Grain Size Upon Isothermal Transformation Behavior of S.A.E. 4140," *Transactions, American Institute of Mining and Metallurgical Engineers*, Vol. 145, 1941, p. 301-314.

15, exhibits a curious "hump", between B and D, which is not a peculiarity of the specimen nor of the method of testing; it is real and is related to the transformation characteristics of the steel.⁷ Fig. 16 shows the microstructure of the end-quenched specimen at the locations designated A, B, C and D in Fig. 15. The microstructure at A, Fig. 16A, consists of martensite with ferrite and a product similar to that formed isothermally at 800 degrees Fahr. (425 degrees Cent.), Fig. 13F. At point B on the curve, the microstructure (Fig. 16B) indicates considerably more transformation than at A and fine pearlite is just beginning to appear. At location C the transformation is not much greater than at B but a much larger portion of the transformed product is fine pearlite, as shown in Fig. 16C. As already noted, in this steel fine pearlite is *harder* than the product formed at slightly lower transformation temperatures, hence the "hump" in the end-quench hardness curve. At point D of the curve, transformation is nearly complete, which accounts for the hardness being lower than at C. G. T. Williams⁸ also reports a similar change in microstructure associated with the hump in an end-quench hardenability curve for a manganese-chromium-nickel steel.

In Fig. 17 cooling curves for points A, B, C and D of the NE 9650 end-quench bar are represented schematically in relation to the isothermal transformation diagram for this steel. The cooling curves shown are not the actual curves for these locations since to truly represent transformation as it occurs on continuous cooling such actual curves would have to be superimposed on a *cooling* transformation diagram rather than an *isothermal* transformation diagram. Rather, the schematic cooling curves in Fig. 17 are based on the microstructures observed in the end-quenched specimens as interpreted on the basis of the isothermal diagram and a knowledge of the resulting transformation products.

As illustrated by the foregoing example, the isothermal transformation diagram and the end-quench hardenability test often serve to supplement one another. The isothermal transformation diagram furnishes a concise and fairly complete picture of the transformation characteristics of the steel, which is useful in planning the heat treatment required to meet a specific need. This type of diagram has

⁷Discussion by E. S. Davenport and R. L. Rickett of TP 1662—"Effect of Several Variables on the Hardenability of High Carbon Steels," by E. S. Rowland, J. Welchner and R. H. Marshall, presented at the February 1944 meeting of the American Institute of Mining and Metallurgical Engineers.

⁸G. T. Williams, "Hardenability Variations in Alloy Steels—Some Investigations with the End-Quench Test," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 157-182.

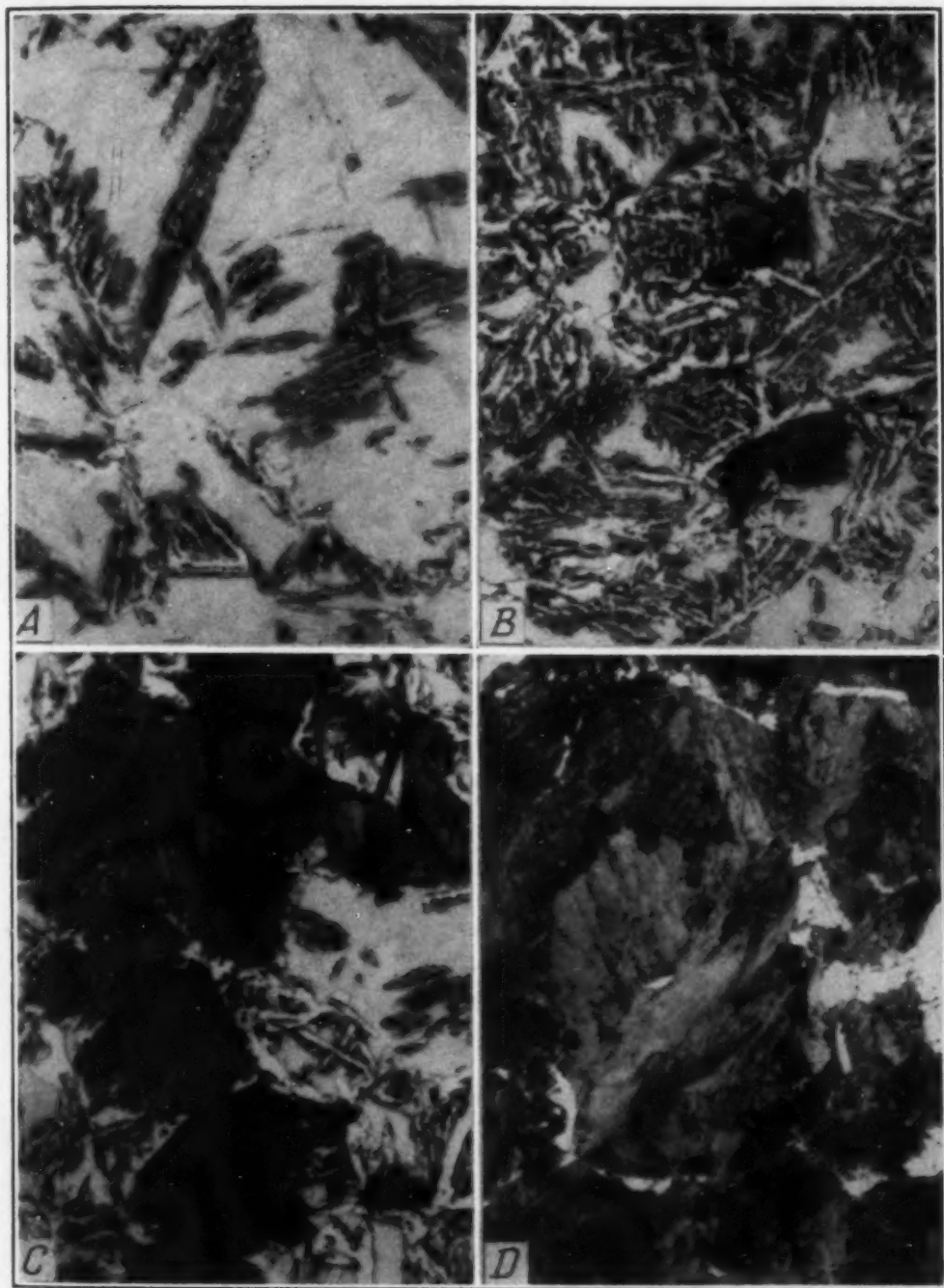


Fig. 16—Microstructure of End-Quenched Hardenability Specimen of NE 9650 at Locations A, B, C and D of Fig. 15. Picral Etch. $\times 1000$.

also been found very useful in interpreting the results of various heat treatments or even results of the hardenability test itself. In addition, the isothermal transformation diagram indicates, as has long been recognized, the hardenability of a steel in a qualitative way; it is not ordinarily a very precise measure of hardenability, however,

because of limited accuracy in determining the short period of time before transformation starts at the "nose" of the diagram for most steels. The end-quench hardenability test, despite its limitations, offers a much more precise *measure* of hardenability than does the isothermal transformation diagram. Naturally, if the end-quench test is to be reliable, it must be carefully conducted and the specimen properly selected so that the surfaces along which hardness is meas-

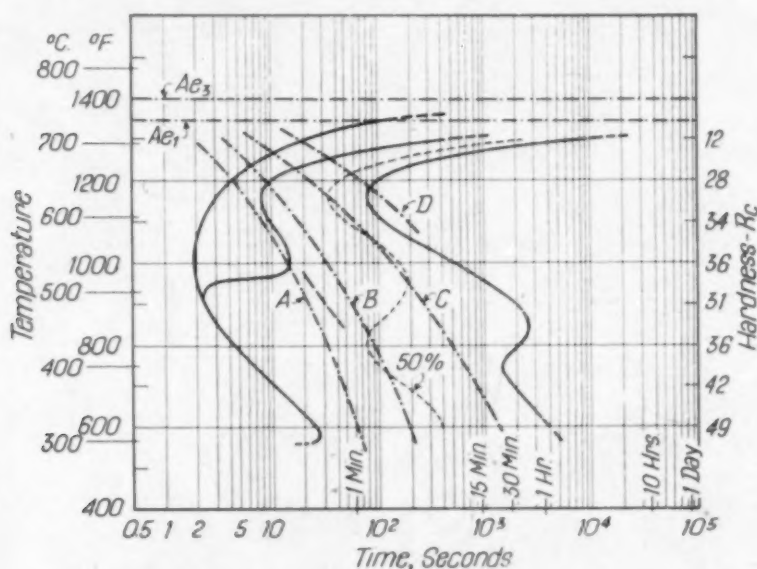


Fig. 17—Schematic Representation of the Cooling of Points A, B, C and D of the NE 9650 End-Quenched Bar (Fig. 15) in Relation to the Isothermal Transformation Diagram for this Steel.

ured are really representative of the steel being tested. It is possible by metallographic examination of the end-quenched specimen to determine the microstructures produced by cooling continuously, as in most actual heat treatments, at any rate within a fairly wide range. This method is worthy of much more general use than at present, since it is the microstructure and not the hardness that is directly related to the properties of most general importance. In many instances, however, the microstructures observed in the end-quenched specimen are difficult to interpret without the information furnished by the isothermal transformation diagram.

It may also be said that the isothermal transformation diagram indicates the general pattern of the heat treating characteristics of a steel. A diagram for one steel of each general type is sufficient for the purpose since this pattern is not greatly affected by minor variations in composition. The precise location of the various portions of

the diagram are, of course, affected by such variations but this is seldom of practical significance since isothermal transformation data are quantitatively applicable only to isothermal heat treatments. Quantitative hardenability, in terms of hardness or microstructures, can best be determined by some direct hardenability test, one of which is the end-quench method. Such a test requires less time to make than is required to determine an isothermal transformation diagram and it could conceivably be made on each lot of steel. If it is to be of any value, however, this test should be conducted in such a way that the result actually indicates the hardenability of the lot and not, for instance, the hardenability at only one location within the original ingot. This feature is one of the more serious limitations of the end-quench test made, as it often is, on bars forged or rolled to nearly the same diameter as the end-quench specimen.

ACKNOWLEDGMENTS

The authors wish to acknowledge the assistance of Mr. J. R. Hamilton in determining some of the isothermal transformation diagrams. They are also grateful for the advice and guidance of Mr. E. S. Davenport, under whose supervision the work was done. Several colleagues in Carnegie-Illinois Steel Corporation were instrumental in initiating part of the work and in supplying some of the steels used.

DISCUSSION

Written Discussion: By L. Luini, metallurgist, Wright Aeronautical Corp., Paterson, N. J.

The authors have presented a very valuable addition to the subject of subcritical transformations in steel and the results will have immediate application in commercial heat treating practices.

Segregation has been pointed out as an influential factor in the determination of reaction rates. The authors have dealt with the banding type of segregation by basing the start of transformation on the first bands to transform and the end of transformation line on the last bands to transform. This I believe will result in a diagram with a beginning line representing a steel of lower alloy content and an ending line representing a steel of higher alloy content than the chemical analysis of the steel indicates. The discrepancy in the reaction rates of different bands will depend upon the degree of segregation in the bands which may or may not be appreciable. I would like to know if the authors found any large difference in the bands with respect to the beginning and ending of transformation. It has been shown by Payson⁹ that ferrite banding

⁹P. Payson, "The Annealing of Steel," *Iron Age*, June and July, 1943.

occurs dependent upon the transformation temperature and is more pronounced at higher transformation temperatures. At 1100 degrees Fahr. (595 degrees Cent.) very little indication of banding could be observed. Is this the type of banding observed by the authors in their work? If it is, my reference above to the significance of the beginning and ending lines of transformation may not be pertinent. However, this is a point which I believe should be brought out.

The macroscopic type of segregation may be eliminated as a variable by choosing that portion of a specimen with a reaction rate intermediate between the maximum and minimum rates observable in the original bar. This leads me to inquire as to the size of the specimen used in the isothermal determinations and the extent of segregation observed by the authors in their bars. The importance of the specimen size factor is augmented when low alloy steels are studied since the transformation is so rapid at about 900 degrees Fahr. (480 degrees Cent.). When a steel begins to transform in less than 1 to 2 seconds at 1400 degrees Fahr. (760 degrees Cent.), it is quite conceivable that specimens, unless sufficiently small, will transform above bath temperature when quenching to temperatures between 1000 and 700 degrees Fahr. (540 and 370 degrees Cent.). This condition will lead to transformation times and hardnesses which are influenced by transformation prior to the time necessary for the specimen to reach bath temperature.

The authors suggest that the suppression of ferrite formation in NE 8949 steel is partially due to the relatively high carbon content. Davenport¹⁰ and similarly Cruciger and Vilella¹¹ have demonstrated that increasing carbon content is primarily effective in retarding the beginning of transformation in the lower temperature levels. It is to be noted that both manganese and molybdenum are relatively high in this NE 8949 steel. Since both manganese and molybdenum are effective in retarding transformation in the higher temperature levels I would like to suggest that the suppression of the ferrite formation is due more to the presence of manganese and molybdenum than to the increase in carbon although the decrease in reaction rate in the lower temperature levels may be primarily due to the carbon content.

The authors are to be complimented on their presentation of the microstructures. It would be interesting and of practical importance to extend this type of work in the direction of correlations with transformations on a falling temperature.

Authors' Reply

The authors want to thank Mr. Luini for his discussion of their paper.

In reply to his questions regarding segregation, considerable of the banding type was present in all the steels investigated, as it is in most commercial alloy steels, and considerable difference in time was required for transformation of different bands. Segregation of this type can be minimized by suitable homogenization of the steel but such treatments were not employed in this investiga-

¹⁰E. S. Davenport, "Isothermal Transformation in Steels," *TRANSACTIONS, American Society for Metals*, Vol. 27, 1939, p. 837.

¹¹J. R. Cruciger and J. R. Vilella, "The Isothermal Transformation of Case-Carburized S.A.E. 4815," *TRANSACTIONS, American Society for Metals*, Vol. 32, 1944, p. 195.

tion because the diagrams were intended to apply to steels as commercially produced.

Davenport¹² has reported the result of homogenization on transformation rate of a limited number of alloy steels and found that the time for beginning and end of transformation may be either longer or shorter than before homogenization. The effect of inhomogeneity of composition upon transformation rate is by no means absent at temperatures below 1100 degrees Fahr. (595 degrees Cent.) and, in these steels, was particularly noticeable after partial transformation at temperatures in the 900 to 1100-degree Fahr. (480 to 595-degree Cent.) range. After complete transformation in this temperature range, the effect of such segregation may no longer be as readily evident as it is after transformation at a higher temperature.

Regarding the possibility of transformation during cooling to the bath temperature, we found no evidence of such transformation in the specimens used, which were 0.040 to 0.060 inch thick.

Mr. Luini suggests that manganese and molybdenum rather than carbon are responsible for slowing the formation of ferrite in NE 8949. While manganese and molybdenum are important factors, carbon is undoubtedly the most important single element in its effect on rate and amount of ferrite formed on transformation.

The authors agree that a correlation of the isothermal data with transformation on continuous cooling is highly desirable. One method of doing this is, of course, the determination of cooling transformation diagrams, which is somewhat difficult to carry out. Much useful information can be gained more simply by correlation of the isothermal transformation diagram with the microstructures of end-quenched hardenability specimens, as discussed in the paper.

¹²Same as reference 3 of paper.

THE EFFECT OF CARBON CONTENT ON HARDENABILITY

By E. S. ROWLAND, J. WELCHNER, R. G. HILL AND J. J. RUSS

Abstract

End quench hardenability determinations were conducted on two base analyses, namely S.A.E. 52XX and S.A.E. 46XX, with the carbon ranging in 0.20 per cent increments from approximately 0.20 to over 1.00 per cent.

Normalized and annealed prior structures were studied for quenching temperatures of 1450 to 1700 degrees Fahr. (790 to 925 degrees Cent.) and time at 1550 degrees Fahr. (845 degrees Cent.) varying from 0 minutes to 4 hours. The data are presented as the effect of carbon content on hardenability for the various conditions considered. The practical aspects of these results as affecting commercial heat treatments are evaluated and discussed.

INTRODUCTION

IN two previous papers the effects of time at quenching temperature, quenching temperature and prior structure were determined for several low and intermediate carbon alloy steels (1)¹ and for three hypereutectoid steels (2). From the results of this work it was postulated that the direction of the change in hardenability with variation in time, temperature and prior structure could be predicted from a knowledge of the carbon content of the steel alone. The extent of the hardenability change was found to depend upon the amount and type of alloy present. The strong carbide formers produced great changes in hardenability as the above three factors were varied while the solution alloying elements caused only comparatively minor variation. It became apparent from this that the hardenability behavior of a given steel was largely dependent upon the exact status of the carbide phase as affecting rate of solution in hypoeutectoid steels and as determining the nucleating effect of the excess carbides in hypereutectoid steels.

¹The figures appearing in parentheses pertain to the references appended to this paper.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The authors are associated with the Metallurgical Department, Steel and Tube Division, The Timken Roller Bearing Company, Canton, Ohio. Manuscript received June 1, 1944.

While there are several general statements in the literature to the effect that hardenability increases with carbon content up to the eutectoid point and decreases with further increase in carbon, no quantitative data are available over the full range of commercial carbon contents when austenitized at normal heat treating temperatures. Digges (3) showed that the critical cooling rate of pure iron-carbon alloys decreases continuously with increase in carbon content under conditions of complete carbon solution, a circumstance which does not occur in commercial treatment of high carbon steels. Cruciger and Vilella (4) determined the S-curves for the core and case of carburized S.A.E. 4815 but again under conditions of complete carbon solution. Their austenitizing temperature of 1800 degrees Fahr. (980 degrees Cent.) is far beyond any commercial hardening temperature for this analysis. Finally, Grossmann (5) in his determination of multiplying factors for calculating hardenability plotted the effect of carbon content at constant grain size up to about 0.85 per cent carbon but stipulated that the values were valid only when complete carbon solution was attained.

In view of the lack of quantitative information capable of accurate interpretation in terms of commercial heat treating conditions, it was decided to investigate the effect of carbon content on the hardenability of two alloy series, as well as the hardenability changes produced by variation in time, temperature and prior structure. One series chosen was based upon S.A.E. 52100 with carbon content ranging to below 0.20 per cent. The other was based upon S.A.E. 4620 with carbon content varying to over 1 per cent. The choice of these series of steels was based upon two reasons. First, they represent widely divergent alloy types, the 52XX series being a strong carbide former while the 46XX series is predominantly a solution alloy base; and second, the base analyses are the two steels most widely used in the anti-friction bearing industry.

EXPERIMENTAL PROCEDURE

Induction heats were melted in approximately 0.20 per cent carbon increments over the range of 0.20 to 1.00 per cent carbon in both alloy series. Since the product of a single 30-pound heat was not sufficient to investigate all conditions, duplicate sets were made and reserved for the effects of time at quenching temperature and quenching temperature, respectively. The chemical analyses are

given in Table I. All heats in the 52XX series were not aluminum treated in conformity with ordinary 52100 practice while all 46XX heats were treated with sufficient aluminum to confer fine-grained properties.

The ingots were hammer forged to 1.25-inch diameter bars, cut in two, one half normalized 1 hour at 1650 degrees Fahr. (900 degrees Cent.) and the other half annealed. Brinell hardnesses and

Table I
Chemical Analyses

52XX SERIES—EFFECT OF TIME								
Heat	C	Mn	P	S	Si	Cr	Ni	Mo
1356	0.18	0.35	0.013	0.024	0.28	1.43	0.27	Nil
1357	0.41	0.37	0.011	0.024	0.34	1.47	0.26	Nil
1358	0.61	0.36	0.012	0.026	0.40	1.48	0.26	Nil
1359	0.81	0.35	0.012	0.023	0.37	1.48	0.27	Nil
1360	0.92	0.35	0.013	0.023	0.37	1.49	0.26	Nil
1412	1.09	0.33	0.011	0.020	0.32	1.59	0.27	Nil
EFFECT OF TEMPERATURE								
1411	0.18	0.32	0.011	0.023	0.28	1.43	0.29	Nil
1409	0.37	0.31	0.011	0.022	0.24	1.46	0.28	Nil
1408	0.58	0.32	0.011	0.023	0.21	1.48	0.28	Nil
1407	0.77	0.33	0.010	0.020	0.30	1.42	0.27	Nil
1406	1.01	0.32	0.010	0.021	0.30	1.45	0.25	Nil
1413	1.07	0.33	0.011	0.022	0.28	1.55	0.27	Nil
46XX SERIES—EFFECT OF TIME								
1371	0.22	0.48	0.010	0.020	0.24	0.22	1.70	0.27
1372	0.43	0.46	0.012	0.022	0.25	0.26	1.75	0.26
1373	0.60	0.50	0.011	0.022	0.27	0.26	1.73	0.27
1374	0.82	0.50	0.012	0.021	0.30	0.24	1.71	0.26
1375	1.03	0.49	0.011	0.021	0.27	0.24	1.70	0.27
EFFECT OF TEMPERATURE								
1401	0.15	0.50	0.010	0.024	0.28	0.25	1.76	0.23
1402	0.29	0.45	0.010	0.023	0.16	0.23	1.74	0.23
1410	0.39	0.47	0.010	0.023	0.24	0.23	1.76	0.23
1403	0.59	0.50	0.010	0.024	0.28	0.23	1.74	0.23
1404	0.79	0.49	0.010	0.023	0.27	0.22	1.76	0.23
1405	1.03	0.48	0.010	0.023	0.24	0.23	1.75	0.24

annealed structures are given in Table II. The intermediate carbon steels were allowed to develop some lamellae in conformity with commercial annealing practice. The normalized prior structures were all typical of small air-cooled bars. Four-inch long, collar-type end quench specimens were machined from the prior treated bars. The heat treating procedure used was the same as previously described (1) and will not be detailed here. Adequate precautions were taken against both decarburization and scaling of the end quench test.

The effect of time at quenching temperature was investigated at intervals of 0, 10, 40 minutes and 4 hours from annealed and normalized prior structures of both alloy series. End quench tests were salt bath heated for the desired intervals at 1550 degrees Fahr.

(845 degrees Cent.), allowing 6 minutes for the specimens to reach temperature. The end quench and hardness testing procedures were in accordance with the A.S.T.M. specifications.

The effect of quenching temperature on the two alloy series from both prior structures was investigated at 1450, 1500, 1550 and 1600 degrees Fahr. (790, 815, 845 and 870 degrees Cent.). The normalized 46XX series was quenched from 1700 degrees Fahr. (925 degrees Cent.) as well, after a 1750-degree Fahr. (955-degree Cent.) normalize.

Table II
Hardness and Structure of Prior Condition

52XX SERIES—EFFECT OF TIME				
Heat	Per Cent Carbon	—Brinell Hardness—		Annealed Structure
		Normalized	Annealed	
1356	0.18	152	111	30% Lamellar—70% Spheroidized + Ferrite
1357	0.41	302	149	50% Lamellar—50% Spheroidized + Ferrite
1358	0.61	302	156	Spheroidized—Trace to 5% Lamellar
1359	0.81	321	170	Spheroidized—Few Dense Carbide Areas
1360	0.92	331	179	Spheroidized—Fine to Medium Carbides
1412	1.09	285	197	Spheroidized—Medium Carbides
EFFECT OF TEMPERATURE				
1411	0.18	149	126	20% Lamellar—80% Spheroidized + Ferrite
1409	0.37	207	149	10% Lamellar—90% Spheroidized + Ferrite
1408	0.58	302	159	Spheroidized—Some Dense Carbide Areas
1407	0.77	331	170	Spheroidized—Fine Carbides
1406	1.01	285	197	Spheroidized—Fine to Medium Carbides
1413	1.07	285	187	Spheroidized—Fine to Medium Carbides
46XX SERIES—EFFECT OF TIME				
1371	0.22	170	149	Spheroidized—Very Fine Carbides
1372	0.43	235	170	50% Lamellar—50% Spheroidized + Ferrite
1373	0.60	262	187	Spheroidized—Trace Lamellar
1374	0.82	321	192	Spheroidized—Few Dense Areas
1375	1.03	321	207	Spheroidized—Fine Carbides
EFFECT OF TEMPERATURE				
1401	0.15	163	149	Spheroidized—Trace to 5% Lamellar
1402	0.29	187	170	20% Lamellar—80% Spheroidized + Ferrite
1410	0.39	229	170	Spheroidized—Some Dense Carbide Areas
1403	0.59	269	187	Spheroidized—Some Dense Areas
1404	0.79	321	187	Spheroidized—Some Dense Areas
1405	1.03	321	207	Spheroidized—Fine Carbides

The specimens were protected in tubes and heated 40 minutes at the quenching temperature in a Hump furnace, allowing an additional 20 minutes for the specimens to reach temperature. Due to the limited amount of material, only one specimen was run for each set of conditions and the average hardnesses of two flats used in constructing the curves. A maximum deviation of 2.5 points Rockwell C was considered an allowable error except in the region of the inflection point where deviations up to 4 points Rc were allowed without duplicating the test.

Fracture grain size was obtained under all conditions of time, temperature and prior structure by quenching $\frac{3}{8}$ -inch diameter by 2-

inch long specimens, fracturing and rating from a set of Shepherd standards. The values given in Table III are the averages of four independent observers. The ratings for the lowest carbon heats in both series at zero time and 1450 degrees Fahr. (790 degrees Cent.) temperature are not reliable as the presence of ferrite causes an apparent coarseness in the fracture.

Table III
Fracture Grain Size

TIME AT TEMPERATURE										
52XX Series										
Heat	Per Cent Carbon	—0 Min.—		—10 Min.—		—40 Min.—		—4 Hours—		
		Ann.	Norm.	Ann.	Norm.	Ann.	Norm.	Ann.	Norm.	
1356	0.18	5	5	6	6	6½	6	6	6
1357	0.41	7½	7½	7	7	7	6½	6½	6
1358	0.61	8½	8½	8	7	8	7	7	6
1359	0.81	9½	9½	9	9½	8½	9½	8	9
1360	0.92	9½	10	9½	9½	9	9½	8½	9
1412	1.09	9½	10	9	9½	9	9½	8½	9
46XX Series										
1371	0.22	9	9	8½	9	8½	9	8½	9
1372	0.43	8½	8½	9	8½	9	8½	8	8
1373	0.60	9	8½	8½	9	9	9	8½	9
1374	0.82	8	8½	7½	9	8	8	8	8
1375	1.03	8½	9	9	8½	8½	9	8½	8½
QUENCHING TEMPERATURE, DEGREES FAHR.										
52XX Series										
Heat	Per Cent Carbon	—1450—		—1500—		—1550—		—1600—		1700
		Ann.	Norm.	Ann.	Norm.	Ann.	Norm.	Ann.	Norm.	Norm.
1411	0.18	5	4½	5½	7	5½	6½	5	6½
1409	0.37	6½	7	6½	6½	6	7	6½	6½
1408	0.58	8½	7½	7½	6½	7	6½	6½	6
1407	0.77	9½	8½	9	9	8	8½	6½	6½
1406	1.01	9½	10	9	9	8½	9	8	8½
1413	1.07	9	9½	9	9½	9	9½	8½	9
46XX Series										
1401	0.15	6½	7	6½	6½	6½	6½	6½	7	7
1402	0.29	7½	8	7	7	7	7	7	7	7
1410	0.39	8½	7½	8	8	8	8	8	7½	8
1403	0.59	8	8	7½	8	7½	7½	7½	8	7½
1404	0.79	8½	9	7½	8½	7½	7½	7	7	6½
1405	1.03	8½	9½	8½	9½	7½	8½	6½	7	4½

PRESENTATION OF RESULTS

Figs. 1 through 4 show the effects of time and quenching temperature variation on all heats of both alloy series from an annealed prior structure. The rate of solution of carbon with increase in time at 1550 degrees Fahr. (845 degrees Cent.) can be followed by observing the progression of maximum hardness at the quenched end of the specimen until, in the higher carbon steels, the hardness reaches 66 Rockwell C. This simple relationship is obscured in the

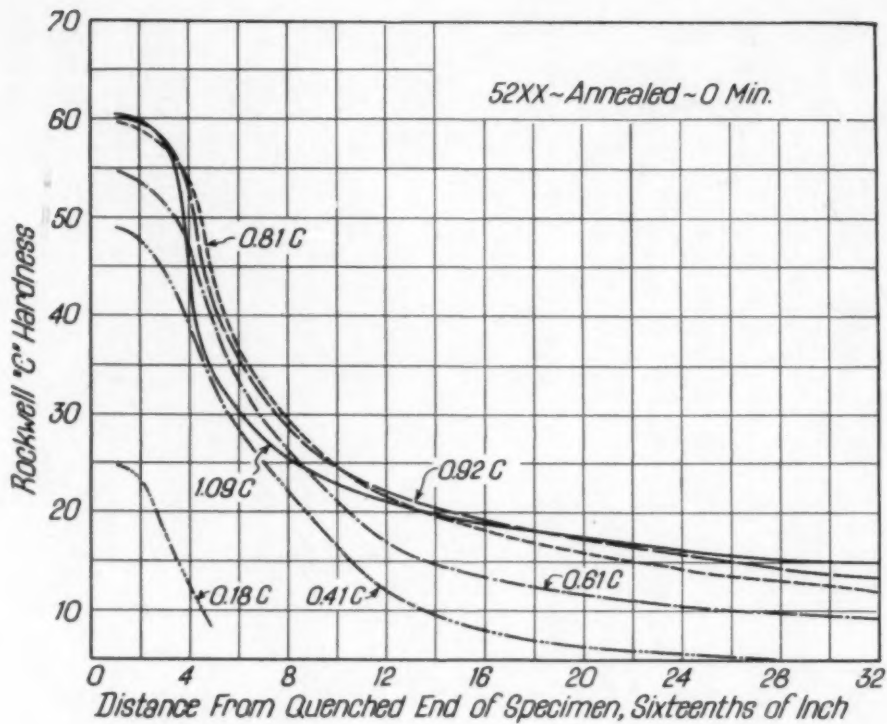


Fig. 1a—Effect of 0 Min. at 1550 Degrees Fahr. on the Hardenability of the 52XX Series from the Annealed Condition.

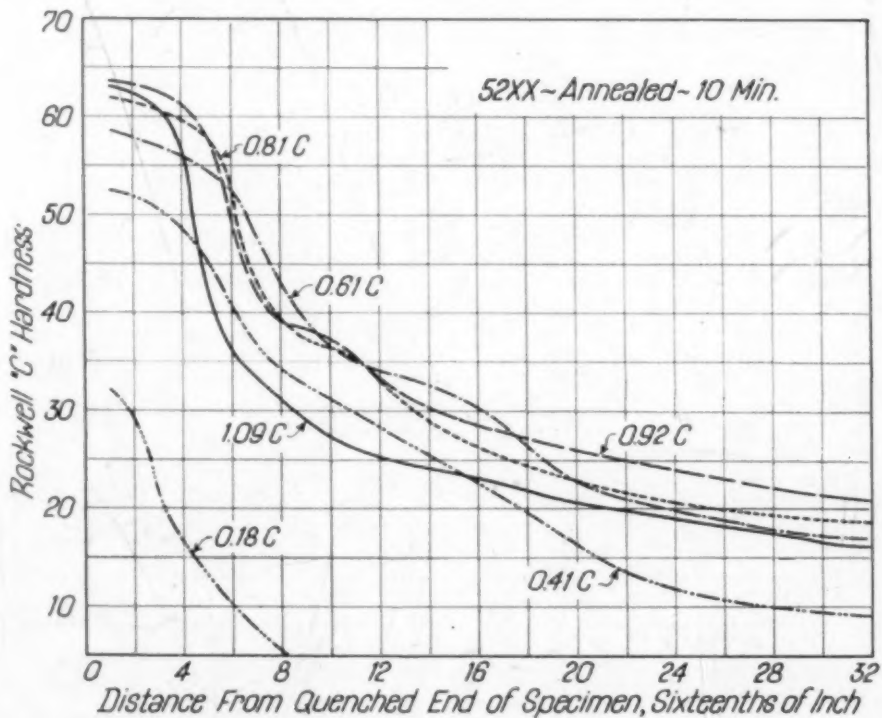


Fig. 1b—Effect of 10 Min. at 1550 Degrees Fahr. on the Hardenability of the 52XX Series from the Annealed Condition.

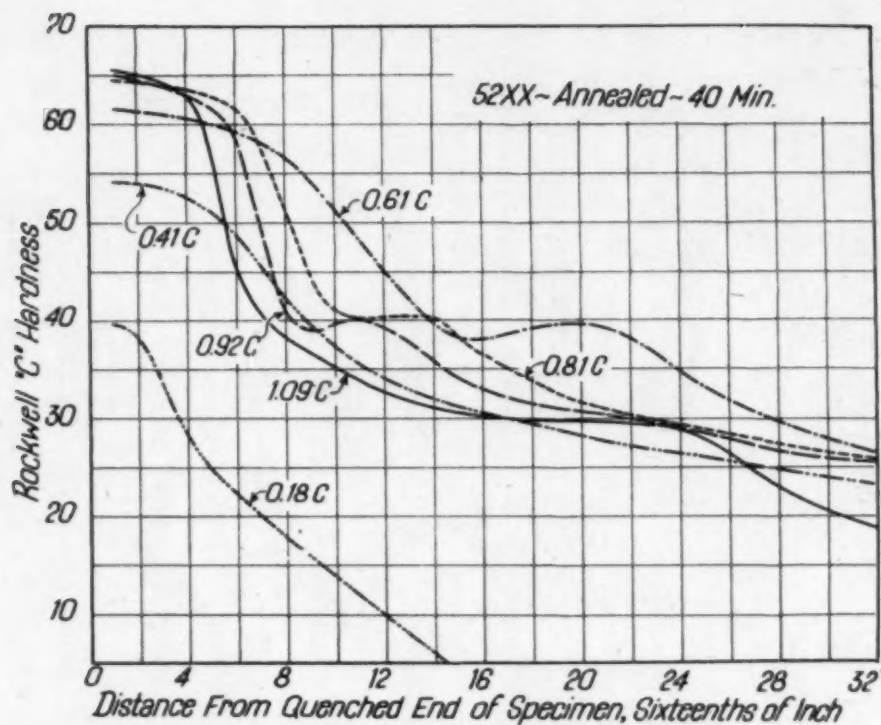


Fig. 1c—Effect of 40 Min. at 1550 Degrees Fahr. on the Hardenability of the 52XX Series from the Annealed Condition.

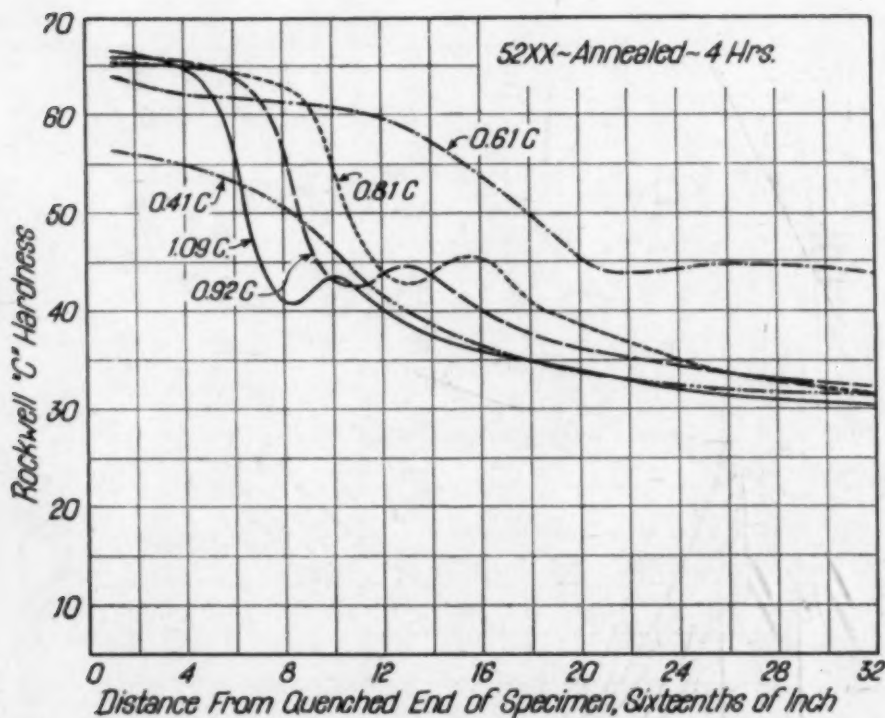


Fig. 1d—Effect of 4 Hrs. at 1550 Degrees Fahr. on the Hardenability of the 52XX Series from the Annealed Condition.

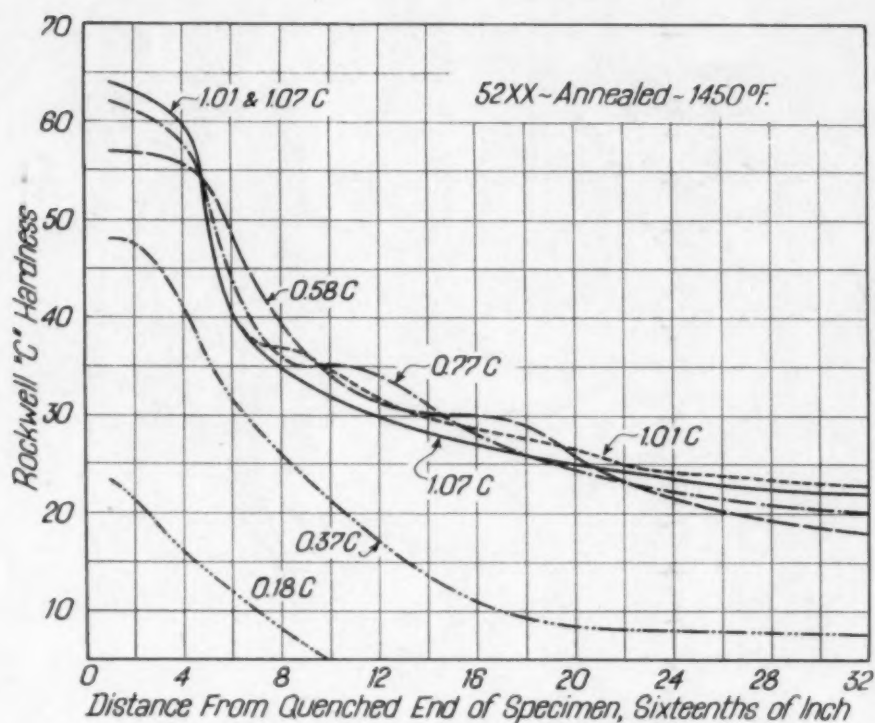


Fig. 2a—Effect of 1450-Degree Fahr. Quenching Temperature on the Hardenability of the 52XX Series from the Annealed Condition.

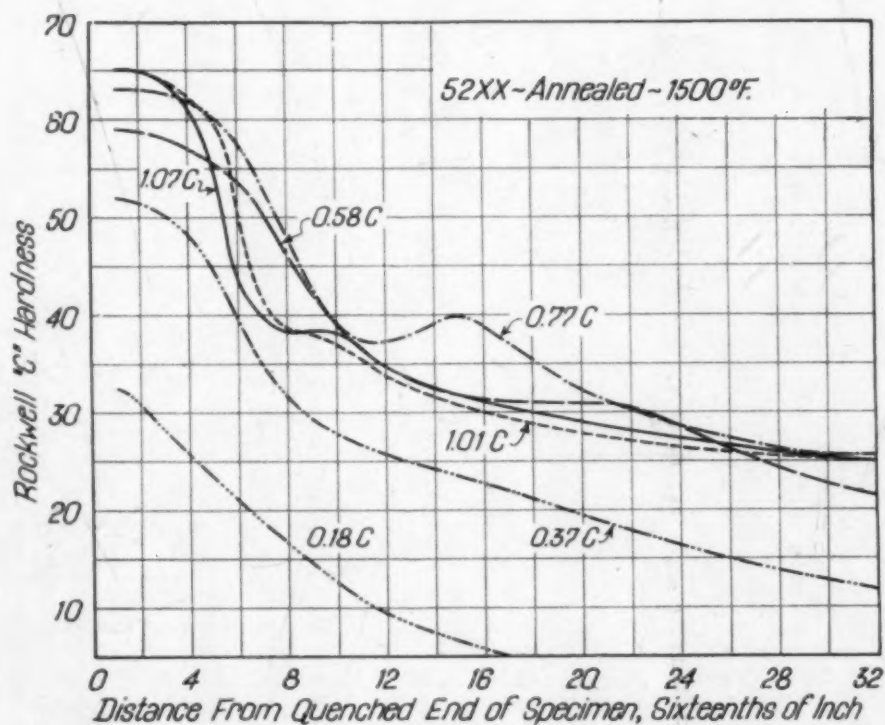


Fig. 2b—Effect of 1500-Degree Fahr. Quenching Temperature on the Hardenability of the 52XX Series from the Annealed Condition.

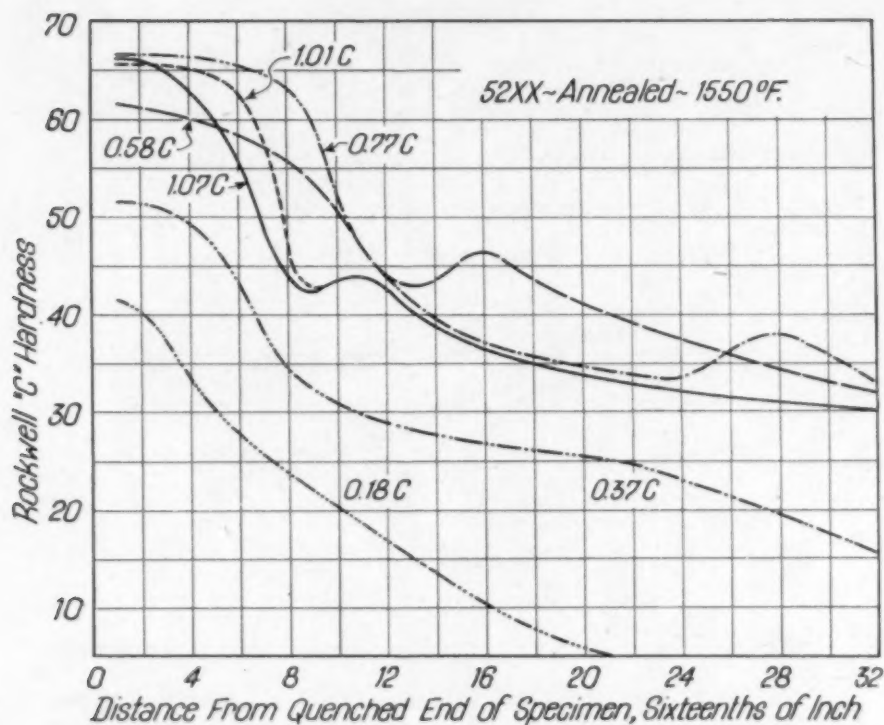


Fig. 2c—Effect of 1550-Degree Fahr. Quenching Temperature on the Hardenability of the 52XX Series from the Annealed Condition.

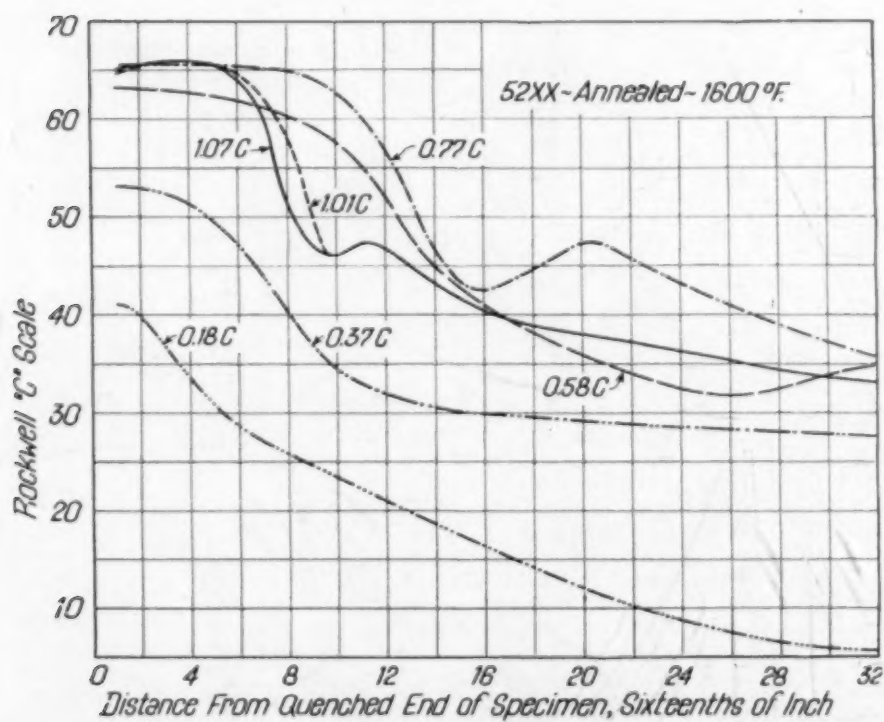


Fig. 2d—Effect of 1600-Degree Fahr. Quenching Temperature on the Hardenability of the 52XX Series from the Annealed Condition.

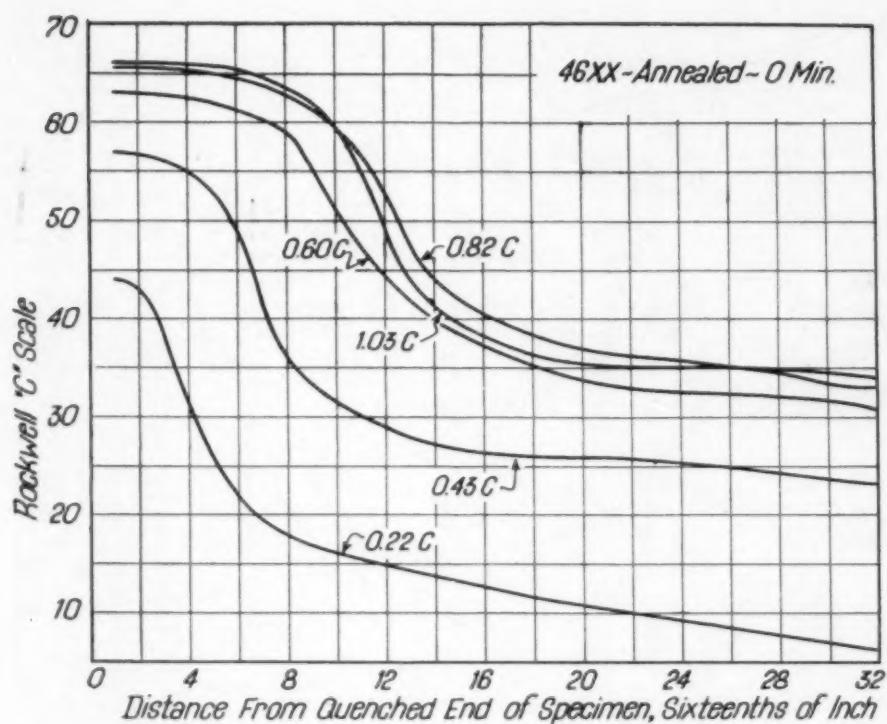


Fig. 3a—Effect of 0 Min. at 1550 Degrees Fahr. on the Hardenability of the 46XX Series from the Annealed Condition.

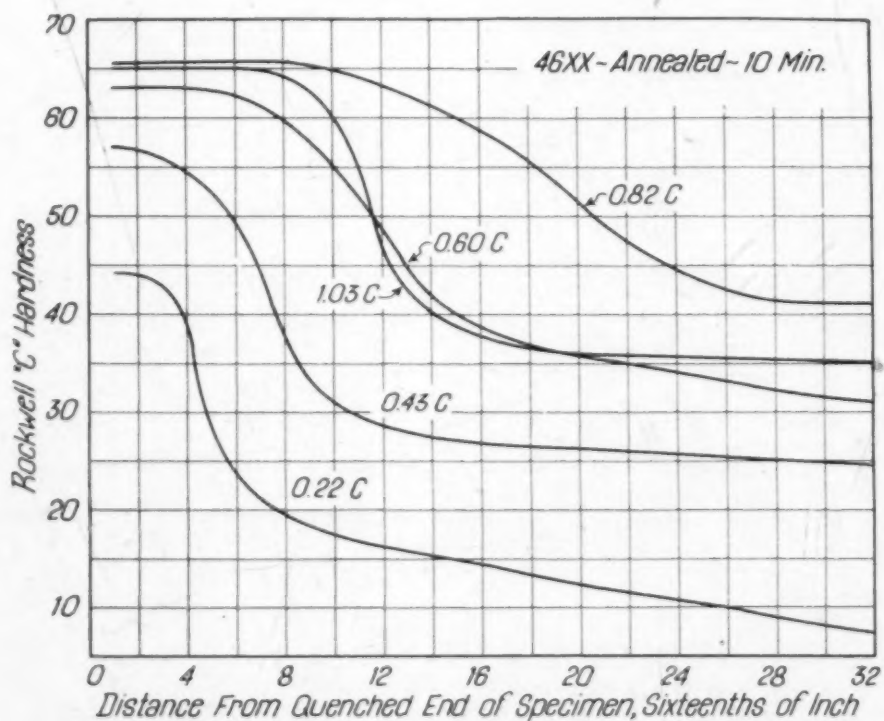


Fig. 3b—Effect of 10 Min. at 1550 Degrees Fahr. on the Hardenability of the 46XX Series from the Annealed Condition.

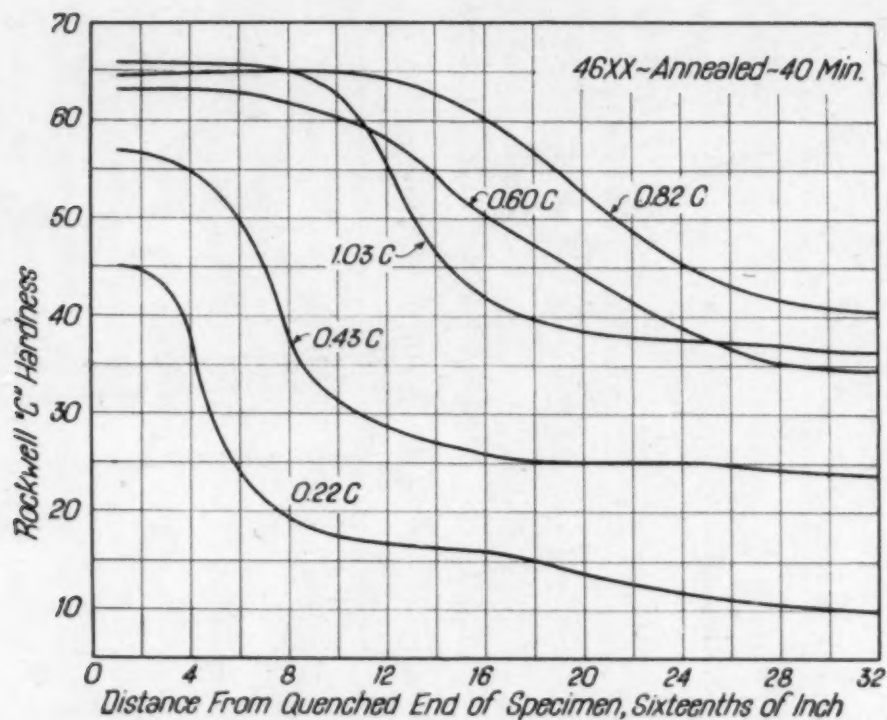


Fig. 3c—Effect of 40 Min. at 1550 Degrees Fahr. on the Hardenability of the 46XX Series from the Annealed Condition.

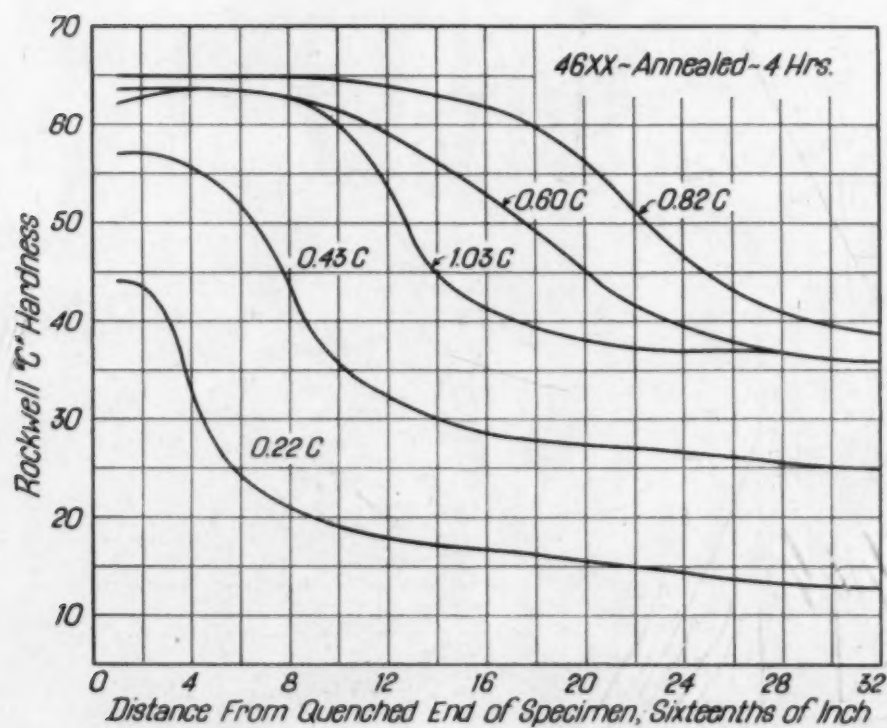


Fig. 3d—Effect of 4 Hrs. at 1550 Degrees Fahr. on the Hardenability of the 46XX Series from the Annealed Condition.

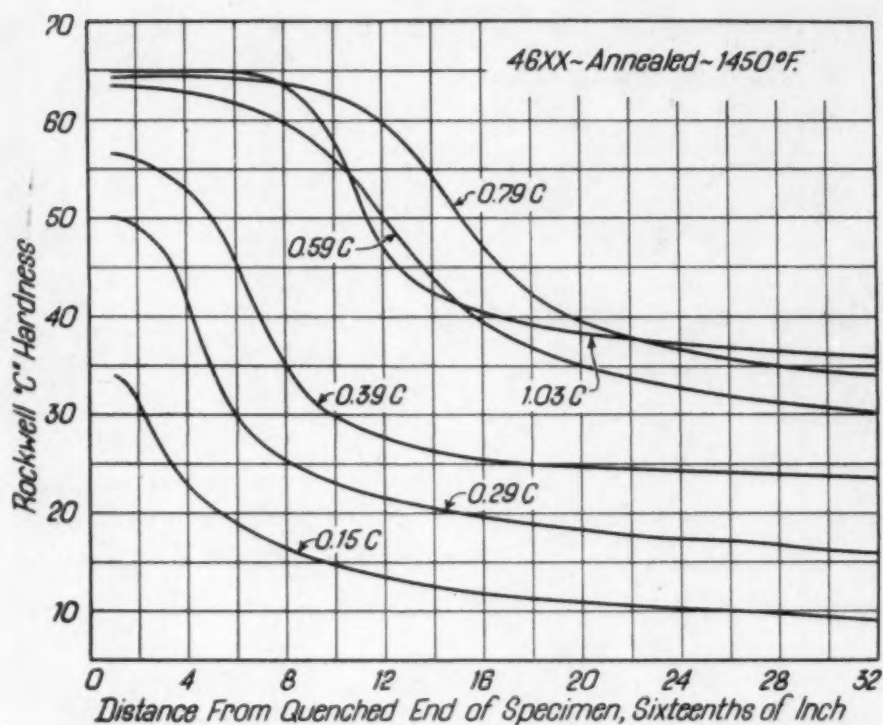


Fig. 4a—Effect of 1450-Degree Fahr. Quenching Temperature on the Hardenability of the 46XX Series from the Annealed Condition.

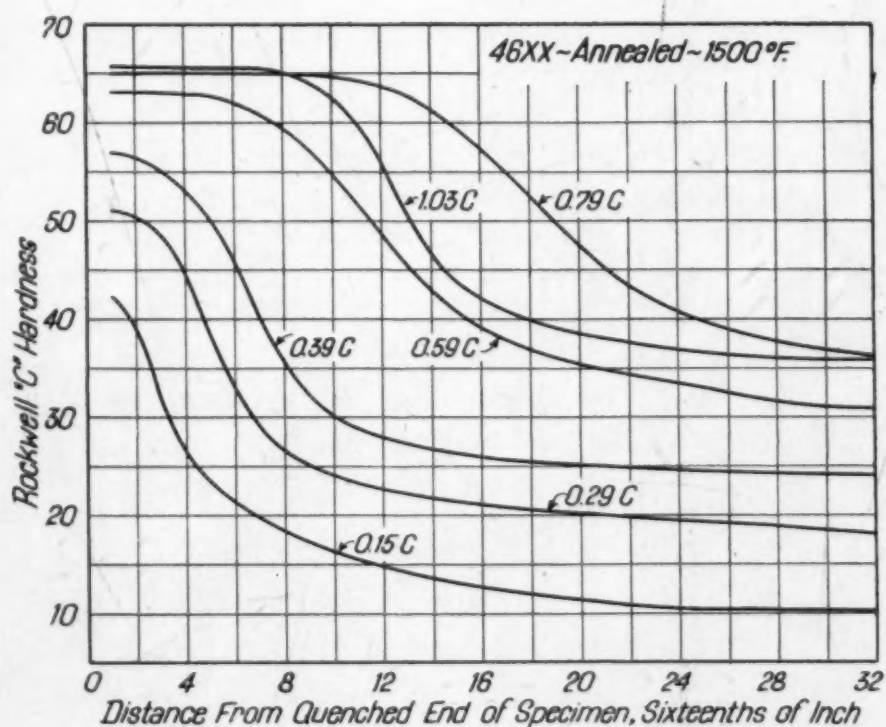


Fig. 4b—Effect of 1500-Degree Fahr. Quenching Temperature on the Hardenability of the 46XX Series from the Annealed Condition.

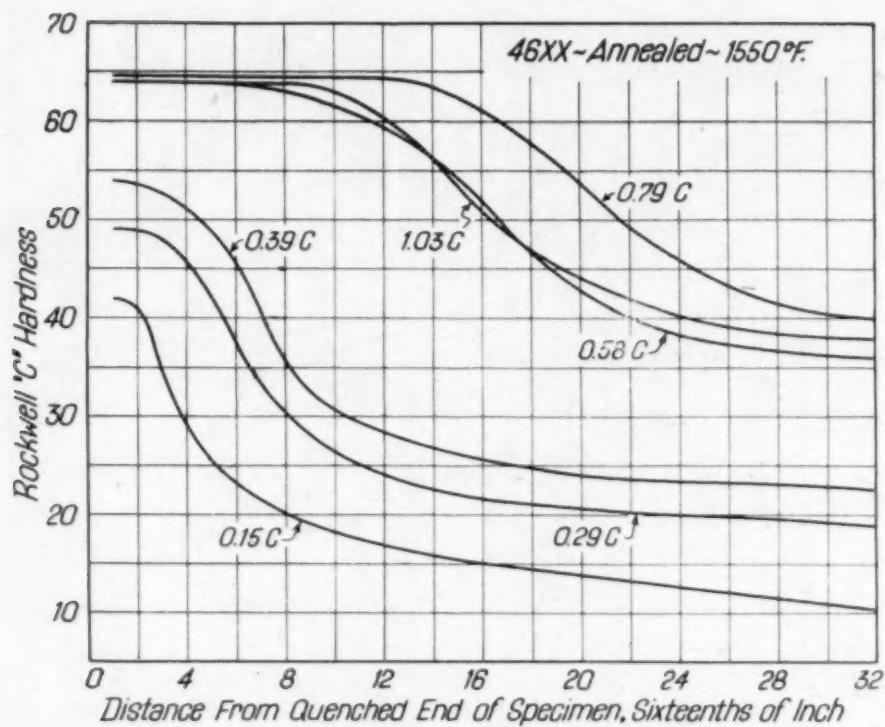


Fig. 4c—Effect of 1550-Degree Fahr. Quenching Temperature on the Hardenability of the 46XX Series from the Annealed Condition.

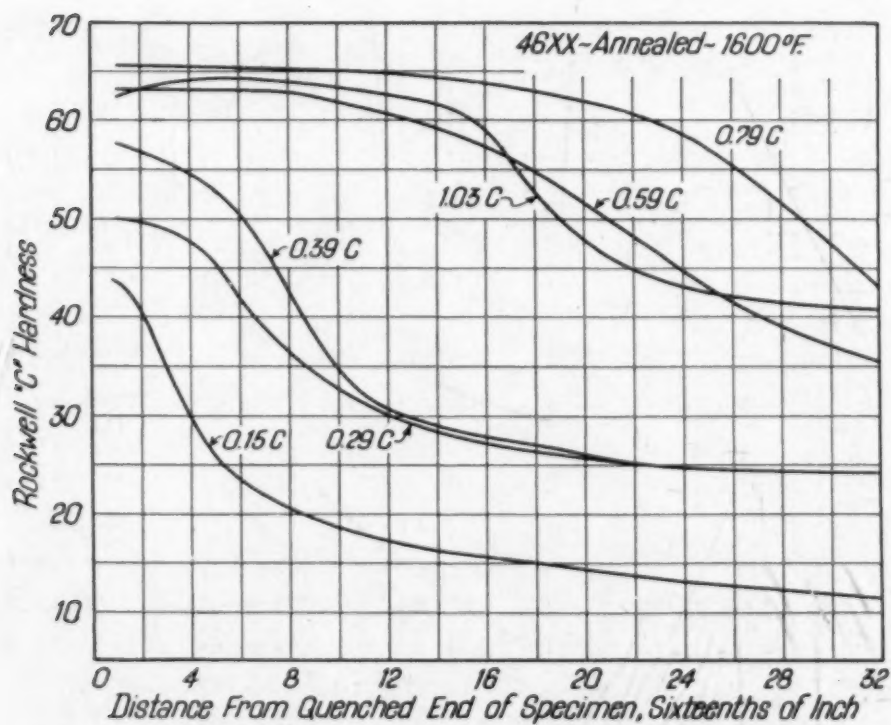


Fig. 4d—Effect of 1600-Degree Fahr. Quenching Temperature on the Hardenability of the 46XX Series from the Annealed Condition.

lowest carbon steels studied by the presence of ferrite at the quenched end at short time intervals. The actual end quench curves for all conditions from a normalized prior state were not included because the maximum hardness at the quenched end was not affected by time at temperature or quenching temperature itself, except for the effect of ferrite solution at low quenching temperatures in the 0.20 per cent carbon steels.

Fig. 5 shows the effect of carbon content on the hardenability of both series as judged by the standard hardenability test—that is, normalized from 1650 degrees Fahr. (900 degrees Cent.) and end quenched from 1550 degrees Fahr. (845 degrees Cent.) after 40 minutes at temperature. The results show clearly, as do all those from annealed prior structures, the rapid increase in hardenability with increasing carbon content to a certain maximum value and the even more rapid decrease in hardenability with further increase in carbon.

In attempting to present in a minimum number of curves the large amount of hardenability data presented here, some criterion of hardenability must be established as a means of evaluating the results. The selection of an arbitrary hardness was not considered warranted because of the carbon content variation. With increase in time at temperature and, to a limited extent, quenching temperature the hardness at 50 per cent martensite increases to a maximum for a given steel. After due consideration, the highest hardness corresponding to 50 per cent martensite at each carbon content and for all conditions of testing was selected as the hardenability criterion. The solid curve of Fig. 6 shows the variation of hardness at 50 per cent martensite with carbon content under conditions of long heating times or high quenching temperatures. The values plotted represent both alloy series and were obtained by microscopic examination of the end-quenched bars and by double differentiation of the end quench curves to obtain an accurate value for the inflection point. The dotted line represents Grossmann's determinations on carbon steels.

Using the solid curve of Fig. 6, the distance from the quenched end of the hardenability bar to the indicated hardness corresponding to the carbon content of the heat was accepted as the measure of hardenability and plotted as a function of carbon content for all conditions of test in Figs. 7 through 10. The effects of time and quenching temperature on the hardenability of the 52XX series are

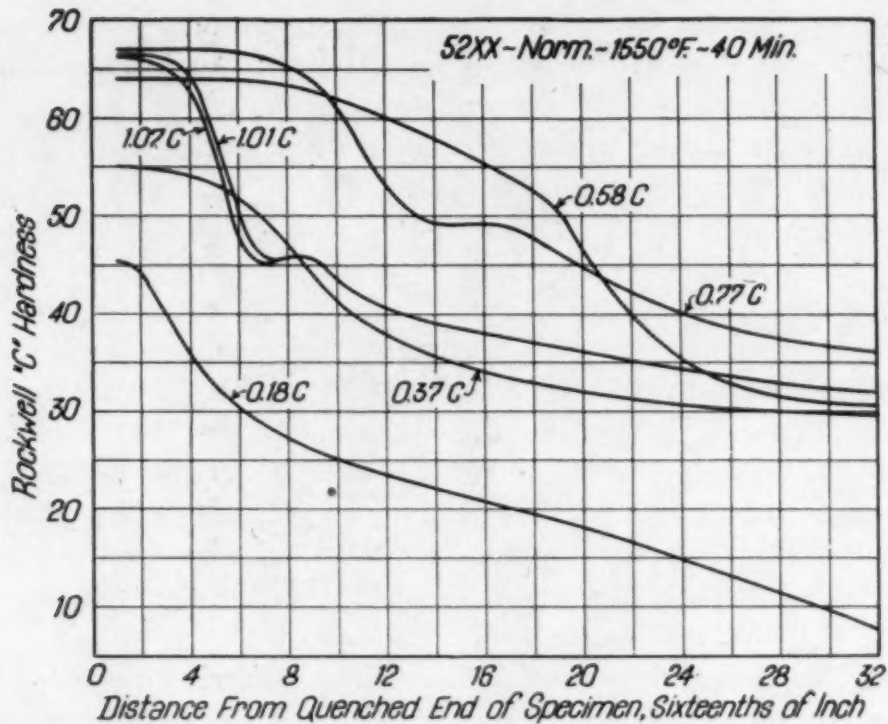


Fig. 5a—Effect of Carbon Content on the Hardenability of the 52XX Series After Heating at 1550 Degrees Fahr. for 40 Minutes from the Normalized Condition.

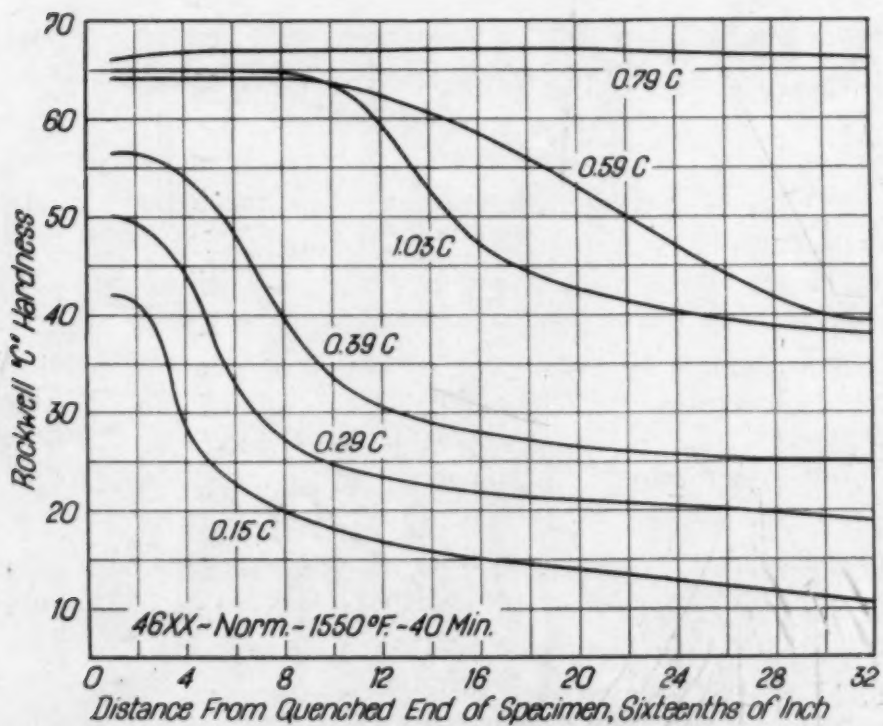


Fig. 5b—Effect of Carbon Content on the Hardenability of the 46XX Series After Heating at 1550 Degrees Fahr. for 40 Minutes from the Normalized Condition.

shown in Figs. 7 and 8, respectively, and comparable information for the 46XX series in Figs. 9 and 10, respectively. In the latter series, the prior normalized 0.80 per cent carbon bars were nearly all air hardening and the hardenability could not be measured by this method. Some index of the behavior in this region can be obtained from Table IV, in which the hardnesses at 2 inches on the bar are given for the various conditions not shown in Figs. 9 and 10.

Table IV
Results of Tests Which Air-Hardened

46XX SERIES—PRIOR NORMALIZED							
Heat	Effect of Per Cent Carbon	Time Condition	Rc Hardness at 2 inches	Heat	Effect of Per Cent Carbon	Temperature Condition Deg. Fahr.	Rc Hardness at 2 inches
1374	0.82	10 min.	59.5	1404	0.79	1500	64
....	40 min.	63	1550	65
....	4 hrs.	63	1600	64
....	1700	65

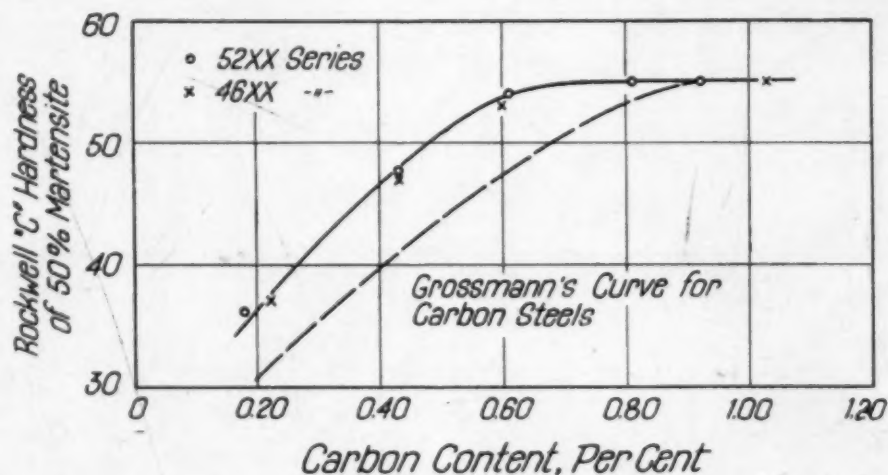


Fig. 6—Hardness of the 50 Per Cent Martensite Structures for Various Carbon Contents.

Since different induction heats were used to investigate the effects of time and temperature in both series, the results were interlocked by virtue of imposing one set of identical conditions for both groups of heats. The values for 40 minutes at temperature (1550 degrees Fahr.) (845 degrees Cent.) should correspond to those at 1550 degrees Fahr. (845 degrees Cent.) of the quenching temperature series (40 minutes). The degree of correspondence is shown in Fig. 11 for both series and both prior conditions and is deemed satisfactory for all practical purposes. The maximum divergence is two sixteenths and occurs at carbon contents where the slope is great and at high hardenability values where the test is most sensi-

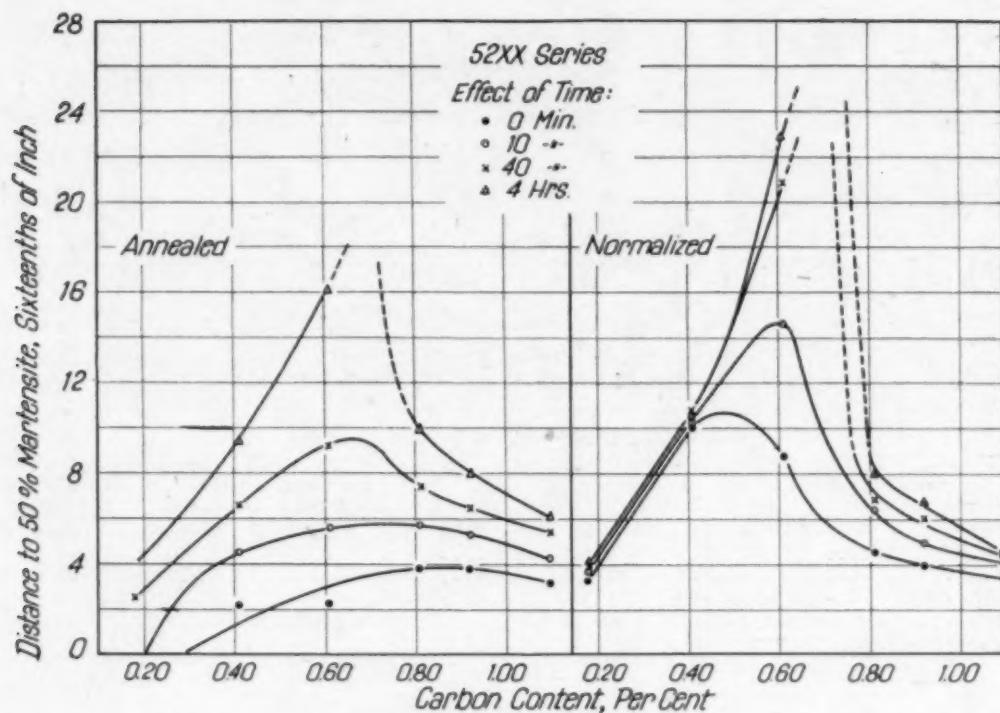


Fig. 7—Effect of Carbon Content on Hardenability of the 52XX Series as Influenced by Time at 1550 Degrees Fahr.

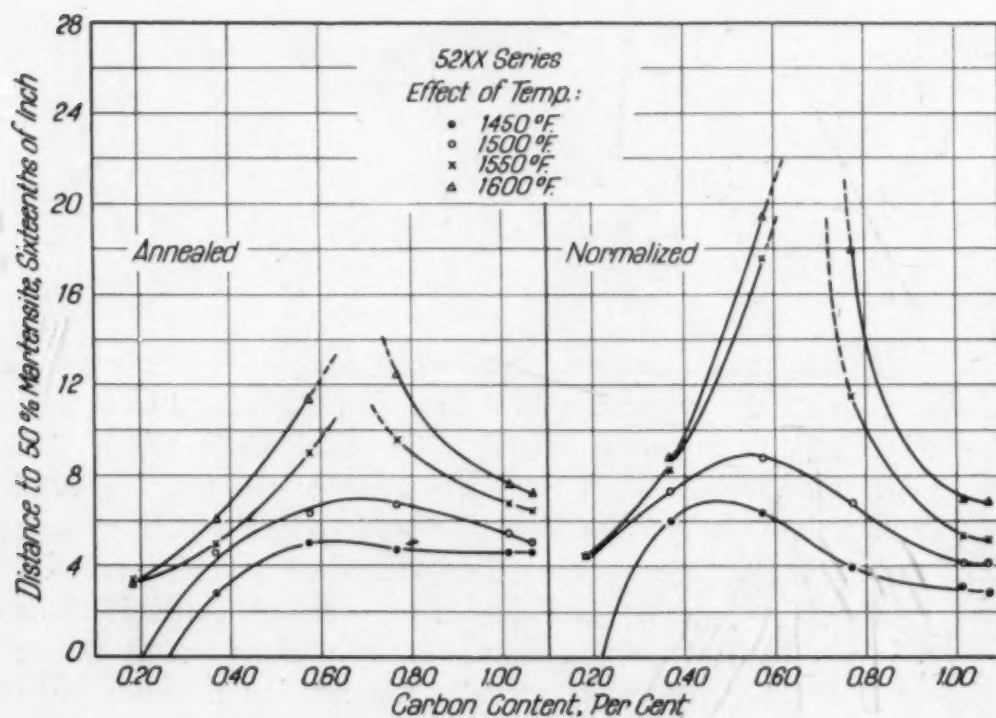


Fig. 8—Effect of Carbon Content on Hardenability of the 52XX Series as Influenced by Quenching Temperature (40 Minutes at Temperature).

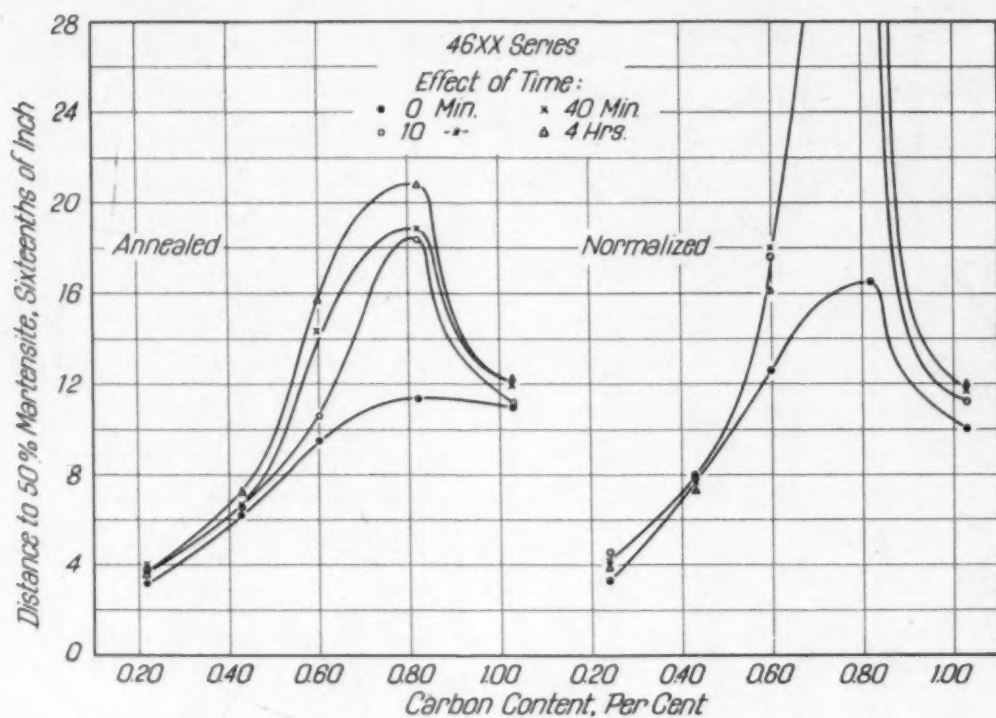


Fig. 9—Effect of Carbon Content on Hardenability of the 46XX Series as Influenced by Time at 1550 Degrees Fahr.

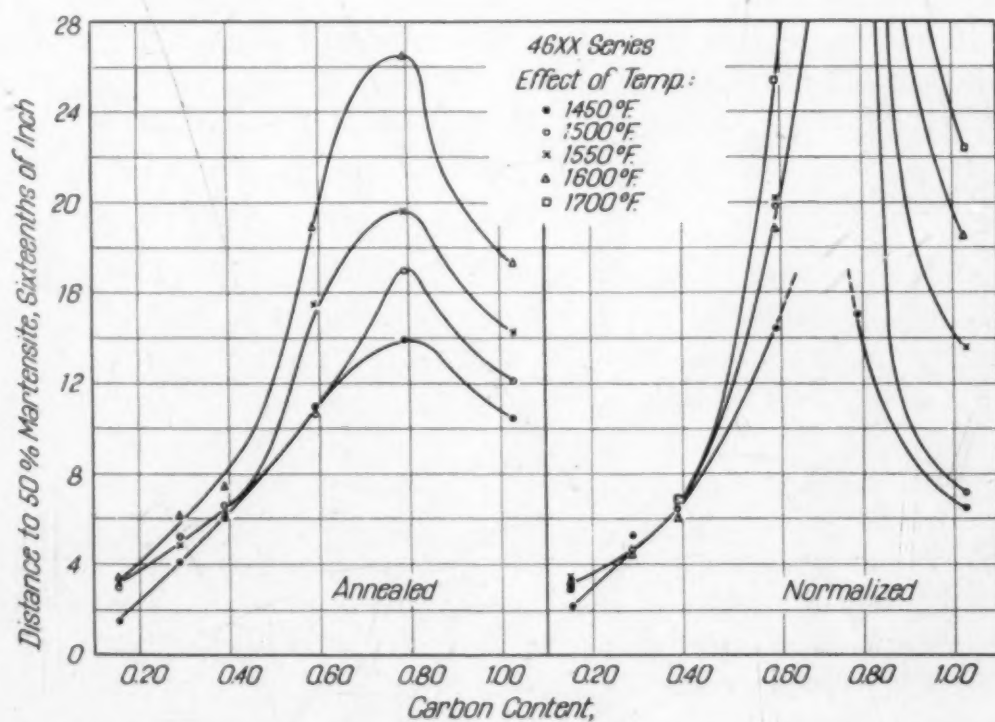


Fig. 10—Effect of Carbon Content on Hardenability of the 46XX Series as Influenced by Quenching Temperature (40 Minutes at Temperature).

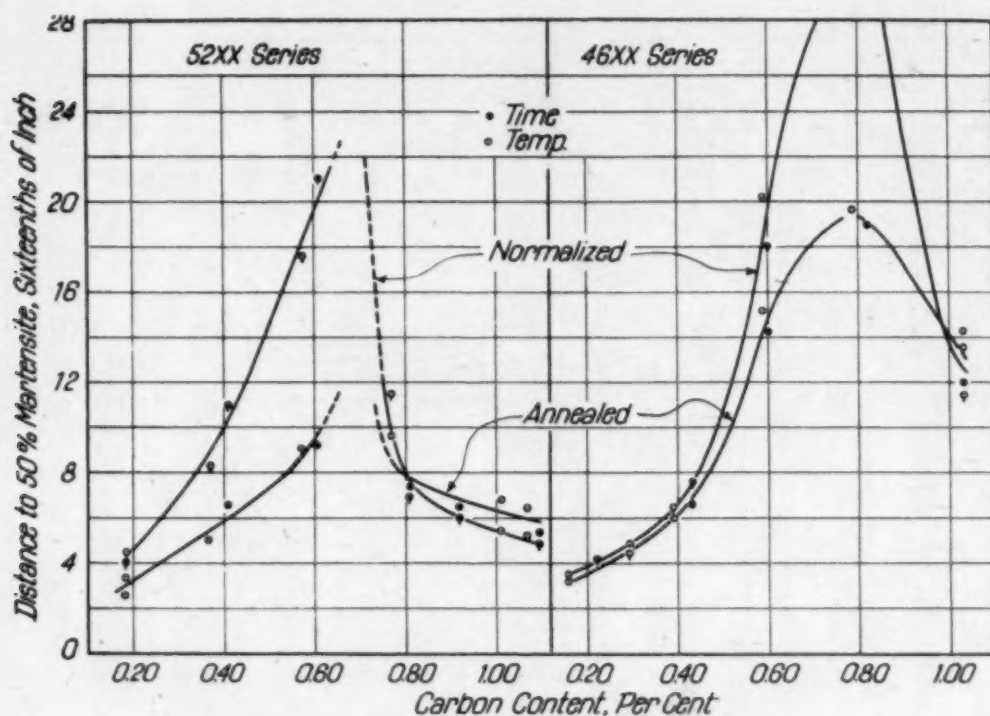


Fig. 11—Comparison of the Effect of Carbon Content on Hardenability of the 52XX and 46XX Series (40 Minutes at 1550 Degrees Fahr.).

tive. The maximum experimental error in the results presented herein is, for this and other reasons, judged to be plus or minus one sixteenth inch. The error is considerably less than this except in the carbon range which produces high hardenability and under conditions of very short times at temperature.

DISCUSSION OF RESULTS

52XX Series—Considering first the 52XX series (Figs. 7 and 8), the hypoeutectoid steels behave with respect to both time and temperature variation exactly as other steels previously investigated (1). The effect of time at temperature from the annealed state is tremendous while negligible from normalized prior structures below about 0.45 per cent carbon. With change in quenching temperature, normalized prior structures reach a substantially constant hardenability somewhat above the A_{c3} temperature over this same carbon range while the annealed condition shows continuous hardenability increase with temperature except at very low carbon contents. As the carbon content increases above about 0.45 per cent, both the hardenability and its variation with time and temperature increase enormously. The hardenability increase in this region concurs with

the grain size change (Table III) but the latter does not explain the difference between the prior structures.

The increments of carbon content used in this investigation were too great to obtain any precise information concerning either the hardenability value or the carbon content at which the maximum in the curve occurs under many conditions of time and temperature. In such cases the curves of Figs. 7 and 8 are dotted beyond the data points to indicate direction and are not continuous in the neighborhood of the maxima. It is possible, however, to arrive at a reasonably close estimate of the carbon content at maximum hardenability by observation of the condition of the carbides in the quenched microstructure at both lower and higher carbon contents.

The composition having maximum hardenability is considered to be the carbon value corresponding to the A_{cm} line at the quenching temperature under conditions of substantial equilibrium with regard to carbon solution and assuming no proeutectoid carbide precipitation during quenching. A carbon content just less than the A_{cm} line at the quenching temperature should show continuous carbon solution with increasing time at temperature and eventually attain complete solution. A carbon content just beyond the A_{cm} line under the same conditions should first show carbon solution and then carbide coalescence as time at temperature is prolonged. For example, the 0.61 per cent carbon prior normalized heat contains fine carbides at zero time at temperature (Fig. 12), less at 10 minutes, just traces at 40 minutes (Fig. 13) and none at 4 hours. The 0.81 per cent carbon heat decreases noticeably in number of carbides up to 40 minutes (Fig. 14) while coalescence occurs at longer times (Fig. 15). All photomicrographs are at 1000 diameters magnification. All specimens were etched in electrolytic sodium picrate.

As a result of these considerations the carbon content at maximum hardenability was estimated to lie in close proximity to 0.70 per cent carbon. While the hardenability value at this maximum is unknown it is suspected of being considerably higher than any compositions investigated. As time at temperature is shortened the carbon content corresponding to maximum hardenability should decrease. This is indicated in Fig. 7 except for short times from annealed prior structures where the extent of carbon solution is quite small.

The same relative conditions obtain for change in quenching temperature at constant time (Fig. 8). From a standpoint of car-

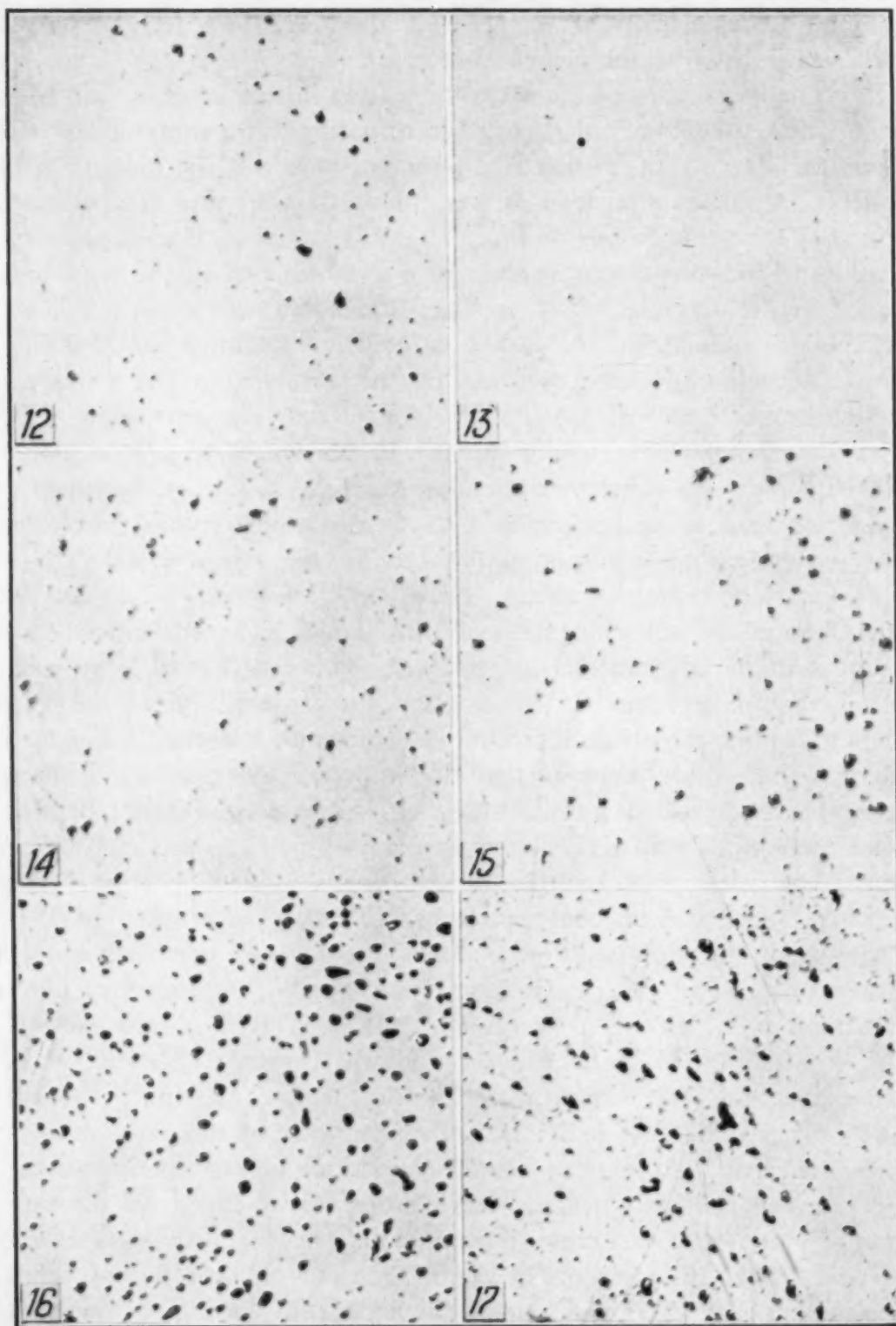


Fig. 12—52XX Series—0.61 Per Cent Carbon, 0 Minutes at 1550 Degrees Fahr.
 Fig. 13—52XX Series—0.61 Per Cent Carbon, 40 Minutes at 1550 Degrees Fahr.
 Fig. 14—52XX Series—0.81 Per Cent Carbon, 40 Minutes at 1550 Degrees Fahr.
 Fig. 15—52XX Series—0.81 Per Cent Carbon, 4 Hours at 1550 Degrees Fahr.
 Fig. 16—52XX Series—1.09 Per Cent Carbon, 4 Hours at 1550 Degrees Fahr.
 Fig. 17—52XX Series—1.09 Per Cent Carbon, 4 Hours at 1550 Degrees Fahr.
 All Magnifications $\times 1000$. All Specimens Etched in Electrolytic Sodium Picrate. Figs.
 12, 13, 14, 15 and 17 Normalized. Fig. 16 Annealed.

bide condition, the 0.58 per cent carbon heat contains no visible carbide after 40 minutes at 1600 degrees Fahr. (870 degrees Cent.) while the 0.77 per cent carbon heat still retains a few carbides after the same treatment. Under these conditions, the maximum was estimated to lie also at about 0.70 per cent carbon. The shift of the maximum to lower carbon contents at progressively lower quenching temperatures is justified by the slope of the A_{cm} line. Grain size is again changing most rapidly with temperature in this carbon range.

The extremely rapid drop in hardenability as the excess carbide increases in amount with further increase in carbon content is obviously due to the nucleating effect of the carbide on austenite decomposition during quenching. The normalized prior state produces the higher hardenability at short times at temperature but at the longer heating times, the annealed condition results in higher hardenability for the hypereutectoid group. With variation in quenching temperature, the annealed state produces equal or superior hardenability at all temperatures, except for the 0.77 per cent carbon heat at the higher temperatures. The total grain size variation with both time and temperature in these hypereutectoid steels is so small that the differences cannot be explained readily on this basis. It was previously observed (2) that in high carbon steels the conditions which produced the fewer visible carbides in the quenched microstructure of a given steel also produced the higher hardenability and this observation has been found to be valid among these steels as well. The microstructures of the annealed and normalized 1.09 per cent carbon heat, quenched after 4 hours at 1550 degrees Fahr. (845 degrees Cent.) are shown in Figs. 16 and 17, respectively, to illustrate this point. It is not deemed valid to suppose that the visible carbides actually act as nuclei for austenite decomposition but it is believed that some definite relationship does exist between the number and distribution of the visible carbides and that of the submicroscopic carbides which actually act as nuclei. The variation in hardenability with change in all factors decreases with increase in carbon content in this range. Experience with other steels has shown that at sufficiently high carbon contents the hardenability is at a minimum and insensitive to all reasonable variations in time, temperature and prior structure.

46XX Series—In comparing Figs. 9 and 10, containing the results of the 46XX series, with Figs. 7 and 8, the resemblance is so striking that little needs to be said except that the low content of

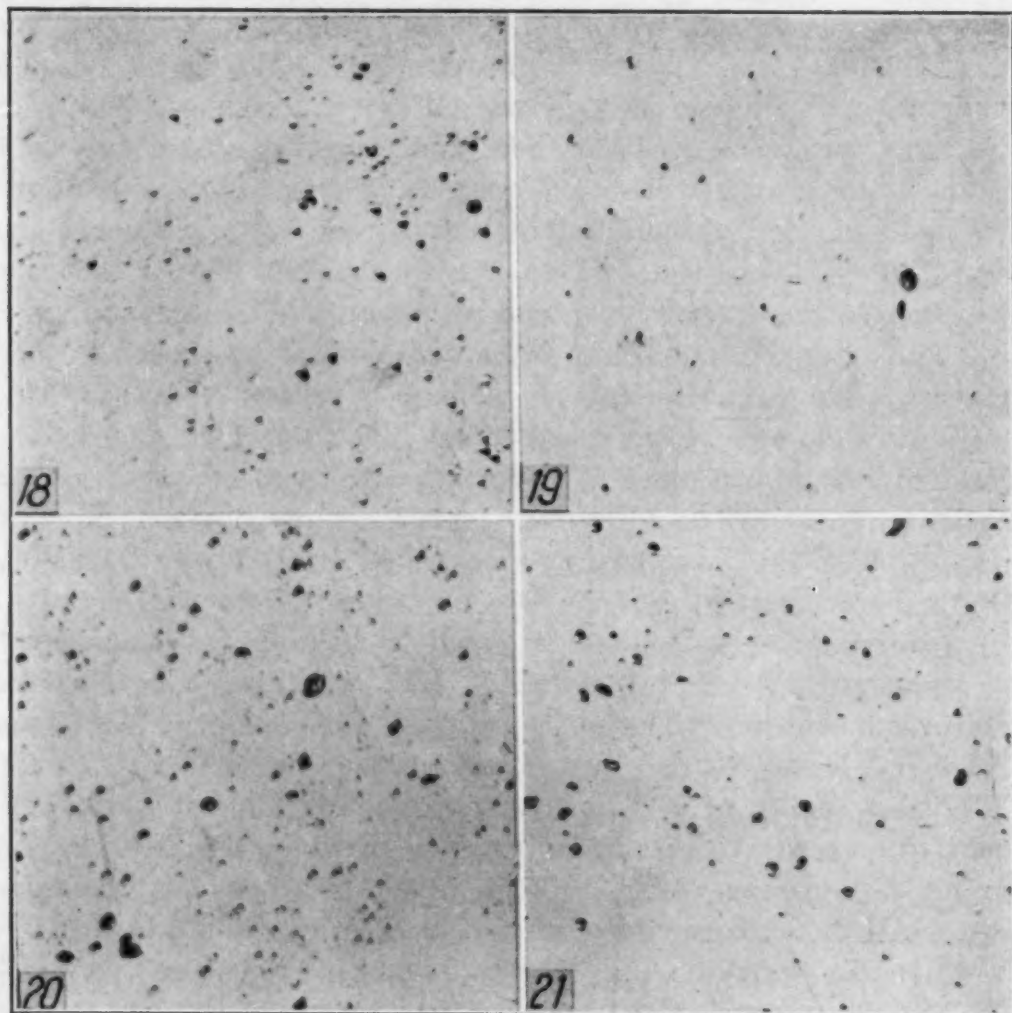


Fig. 18—46XX Series—0.82 Per Cent Carbon, 10 Minutes at 1550 Degrees Fahr. (Annealed).

Fig. 19—46XX Series—0.82 Per Cent Carbon, 40 Minutes at 1550 Degrees Fahr. (Annealed).

Fig. 20—46XX Series—1.03 Per Cent Carbon, 10 Minutes at 1550 Degrees Fahr. (Annealed).

Fig. 21—46XX Series—1.03 Per Cent Carbon, 4 Hours at 1550 Degrees Fahr. (Annealed). All Magnifications $\times 1000$. All Specimens Etched in Electrolytic Sodium Picrate.

carbide-forming elements in this series renders the heats much less sensitive to variation in time and quenching temperature and the maximum hardenability occurs at a higher carbon content because of the different alloy content. In this latter connection, the carbide condition existing in the quenched microstructure of the 0.82 and 1.03 per cent carbon prior annealed heats after different intervals of time at temperature can be judged from Figs. 18 through 21. After 10 minutes at temperature (Fig. 18) the 0.82 per cent carbon heat shows just a few fine carbides while 40 minutes (Fig. 19) reduces

this to a trace and after 4 hours none are visible. The 1.03 per cent carbon heat after 10 minutes (Fig. 20) at temperature shows fine carbides which coalesce noticeably at the 4-hour interval (Fig. 21). Under conditions of practical equilibrium at 1550 degrees Fahr. (845 degrees Cent.), the carbon content corresponding to maximum hardenability in this series probably lies only slightly above 0.82 per cent carbon.

A further point of difference between the two series is the greater increase in hardenability of the 46XX series with increase in quenching temperature in the hypereutectoid carbon range. The grain size change is in accordance with this difference as shown in Table III. It is considered inevitable that further increase in carbon would rapidly converge the hardenability values at the several quenching temperatures but neither time nor manpower were available to add additional heats in this range.

It is of considerable interest to note that substantially equal hardenability exists between the two series up to approximately 0.60 per cent carbon content when the effect of rate of carbon solution is minimized—that is, when the comparison is made between normalized prior structures. This is contrasted sharply by the rapid divergence as the carbon increases beyond 0.60 per cent and when 1.00 per cent carbon is reached the hardenability of the 46XX steels (when based on cooling rates) is several times that of the 52XX type. Such results are not entirely unexpected when one reviews the functions of the predominant alloying elements in each series. These results further point out that while a carbide forming and a solid solution element impart a given hardenability characteristic to a base analysis in the lower carbon ranges this is indeed no criterion as to what these same elements may do in the same type steel of higher carbon content.

The differences pointed out above are ones of degree only and do not affect the fundamental relationship between carbon content and hardenability. As a result of the close parallelism of the behavior of these two widely divergent alloy series with regard to hardenability change with carbon content and with variation in the related factors of time, temperature and prior structure, it is believed that the direction of the hardenability change of a given steel can be predicted with confidence from a knowledge of its carbon content alone, while the extent of the deviation will depend upon the type and amount of alloying elements present.

The behavior of both these series emphasizes the difficulty of obtaining reproducible results from the carburized end quench test developed by Jominy and Boegehold (6) to measure case hardenability. These results show that a variation of a few points in carbon at the level of hardness measurement has a much greater influence on hardenability than a much greater alloy change. The control of carbon content in carburizing poses a considerable problem in performing this test with reasonable reproducibility.

PRACTICAL CONSIDERATIONS

Several points of interest from a standpoint of practical heat treatment of steels in these and related series deserve mention. In the case of high carbon steels where full carbon content is often desired on the working surface, extreme care must be taken to prevent decarburization in hardening because neither a hardness test, a file test nor an etch test for soft spots can be expected to detect the presence of a considerable amount of partial decarburization on the surface. Since the hardenability of decarburized areas in the range of from about 0.60 per cent carbon to the full carbon content of the steel will all be higher than that of the base analysis, these areas will be fully martensitic in hardening if the quench is sufficiently vigorous to harden the base material. Therefore the degree of partial decarburization detectable by the file test, for example, will more often depend upon the hardness of martensite as controlled by its carbon content than upon hardenability of the decarburized areas. If these two series of steels are any criterion, quite a considerable degree of partial decarburization may be present before quenching pearlite appears on the surface if it be absent at the full carbon level below the surface.

In considering the hardening of carburized parts, these results would seem to indicate that the file-soft skin sometimes found on the surface of pack carburized and pot-quenched alloy steel parts may be due to excessively high carbon content on the extreme surface rather than to decarburization. For effective oil quenching of carburized low alloy steels, at least, it would certainly appear logical to avoid high maximum carbon concentrations if freedom from quenching pearlite on the extreme surface is to be prevented.

These results offer a ready explanation of the general observation that soft spots on carburized and hardened work are of only

shallow penetration and rarely extend more than one-third of the depth of hard case. If parts are slightly decarburized in reheating for hardening, soft spots present in the highest carbon subsurface layer will be masked from either a file or an etch test by the fully hardened decarburized layer. This condition can be readily observed by carburizing an end quench bar, reheating it to allow partial decarburization to occur, and end quenching. If a 0.015-inch deep longitudinal flat on the bar be polished, etched and examined microscopically the full effects of the carbon content variation are apparent.

SUMMARY AND CONCLUSIONS

This investigation was conducted on a group of induction heats varying in carbon content in approximately 0.20 per cent increments from 0.20 per cent to over 1 per cent carbon in each of two base analyses: S.A.E. 52100 and S.A.E. 4620. End quench hardenability tests were run on bars from these heats from two prior structures, annealed and normalized, and the effects of variation in time at quenching temperature up to 4 hours and quenching temperatures up to 1700 degrees Fahr. (925 degrees Cent.) determined. The hardenability results were plotted as a function of carbon content under the conditions investigated for both alloy series and the practical application of the data to commercial heat treatment considered.

The following conclusions were drawn:

1. At normal hardening temperatures, the hardenability of a given alloy series increases with carbon content to a maximum value corresponding to the position of the A_{cm} line under the conditions of testing imposed and then decreases with further increase in carbon content. This decrease is due to the nucleating effect of the excess carbide on the decomposition of austenite on quenching.
2. The similarity in hardenability behavior between the two alloy series was so marked that it is believed that the direction of this behavior under the influence of change in time, temperature and prior structure can be predicted with some confidence from a knowledge of the carbon content alone. The type and amount of alloying elements control the extent of the hardenability change, carbide formers producing the maximum effect and solution elements the minimum.

3. In commercial hypoeutectoid alloy steels, annealed prior structures increase in hardenability only slowly with increase in time at normal quenching temperatures while normalized prior structures assume maximum hardenability at very short time intervals. The latter are substantially insensitive to quenching temperature variation between the A_{c3} and the coarsening temperature while the former increase continuously in hardenability with increase in quenching temperature.

4. With normal commercial soaking times and quenching temperatures, hypereutectoid steels produce maximum hardenability from annealed prior structures rather than from normalized. This is believed to be due to and controlled by the number and distribution of those excess carbides which act as nuclei for austenite decomposition.

5. In the eutectoid carbon range, the hardenability change is enormous with variation in conditions but, in general, obeys the same laws as in lower carbon ranges. The normalized prior structure will produce higher hardenability than the annealed under all reasonable conditions of time and quenching temperature.

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5. M. A. Grossmann, "Hardenability Calculated from Chemical Composition," *Transactions, American Institute of Mining and Metallurgical Engineers*, Vol. 150, 1942, p. 227.
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DISCUSSION

Written Discussion: By Thomas G. Digges, metallurgist, U. S. Department of Commerce, National Bureau of Standards, Washington, D. C.

The authors state that there is a lack of quantitative information on the effect of carbon, over the range used commercially, on the hardenability of steels when austenitized at normal heat treating temperatures. In 1938,¹ the writer described the preparation of high-purity iron-carbon alloys and plain carbon steels and presented data showing how carbon affected their hardenability. The method used for preparing these materials eliminated variables other than carbon in the composition. The relations of the critical cooling rate to carbon content of the alloys and steels are summarized in Fig. A of this discussion. For

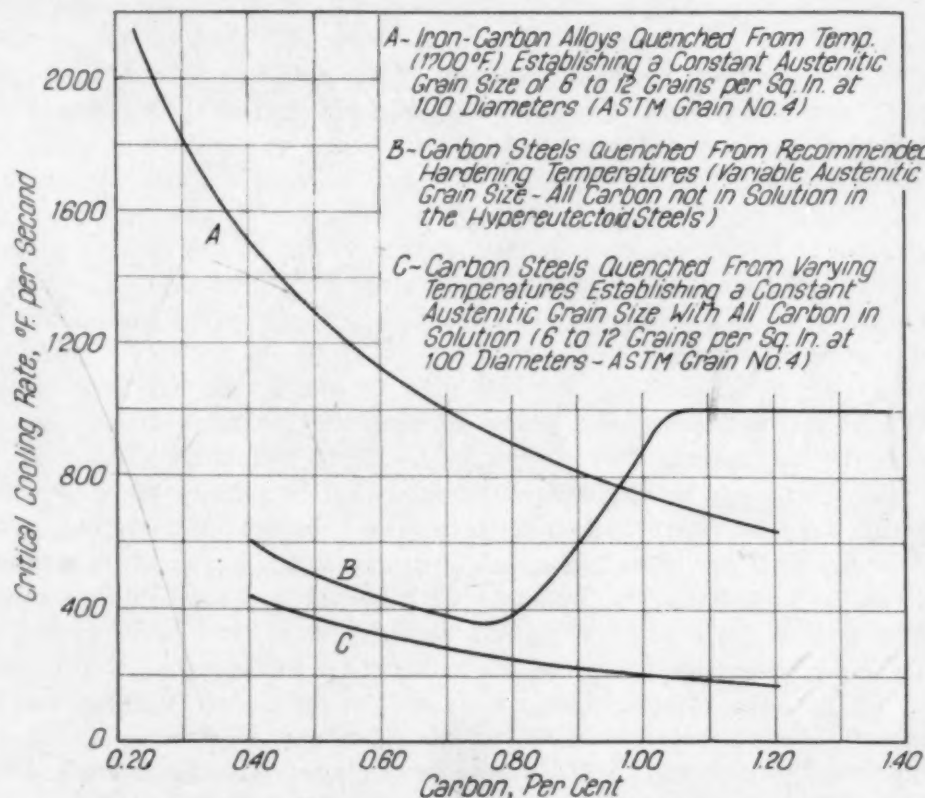


Fig. A—Relation of Critical Cooling Rate to Carbon Content of the Iron-Carbon Alloys and Carbon Steels. The critical cooling rate was taken as the average cooling between 1110 and 930 degrees Fahr. (600 and 500 degrees Cent.) which produced in the quenched specimens a structure of martensite with nodular troostite (fine pearlite) in amounts estimated to be between 1 and 3 per cent. The base composition of the plain carbon steels consisted of 0.46 per cent manganese, 0.043 per cent phosphorus, 0.025 per cent sulphur, 0.15 per cent silicon, 0.02 per cent chromium, trace of vanadium, and 0.11 per cent nickel.

the conditions of constant austenite grain size and complete solution of carbon, the critical cooling rate decreased (hardenability increased) continuously with

¹Thomas G. Digges, "Effect of Carbon on the Critical Cooling Rate of High-Purity Iron-Carbon Alloys and Plain Carbon Steels," Research Paper RP1092; *Journal of Research*, National Bureau of Standards, Vol. 20, p. 571, May 1938.

increase in carbon of both the high-purity alloys (curve A) and the plain carbon steels (curve C).

For the carbon steels quenched from the usually recommended hardening temperature (curve B), the critical cooling rate decreased with increase in carbon up to about 0.75 per cent and thereafter increased rapidly with increase in carbon up to about 1.05 per cent. Further increase in carbon from 1.05 to 1.40 per cent had no appreciable influence on the critical cooling rate. Thus, the steel of about eutectoid composition had the minimum critical cooling rate, or was deeper hardening than either the steels of hypoeutectoid or hypereutectoid compositions. However, these critical cooling rates (curve B) were affected by variations in grain size and carbon content of austenite as well as by total carbon. With the steels ranging in carbon from about 0.40 to 0.75 per cent the average grain size was approximately constant and all carbon was in solution at the time of quenching. The difference in their hardenability was caused by a difference in the carbon content of the austenite. With carbon in excess of about 0.80 per cent, the carbon content of the austenite (dissolved carbon) remained constant (quenching temperature of 1425 degrees Fahr.) but differences existed in the undissolved or total carbon and presumably in grain size. The rapid increase in critical cooling rate with increase in carbon from about 0.80 to 1.05 per cent was due principally to variations in the number of free carbides but a further increase in the number or size of the carbide particles was without effect on the critical cooling rate or hardenability. The authors in their study of the hardenability of certain alloy steels by the end quench test find this same relationship.

None of the conventional methods tried by the writer has been entirely satisfactory for delineating the grains of austenite in high carbon steels at temperatures regularly used in hardening. Under such conditions, the free carbides tend to coalesce as spheroids not only at the boundaries but also within the grains and the parent austenite grains are obscure after cooling either slowly or rapidly. The writer, however, is inexperienced in rating the austenite grain size by comparing the fractures of hardened specimens with standard fractures such as those prepared by Shepherd. The authors have pointed out that the presence of free ferrite causes an apparent coarseness in the fracture. Would the presence of spheroidized carbides tend to make the appearance of the fracture finer or coarser than the actual austenite grain size?

Written Discussion: By C. P. Sun, investigator, Main Laboratory, Bethlehem Steel Co., Bethlehem, Pa.

I read this paper with great interest and wish to congratulate the authors for their splendid work. There is one point that I would like to discuss with the authors.

The authors found that the hardenability of a given alloy series increases with carbon content to a maximum value and then decreases with further increase in carbon content. They conclude that the decrease is due to the nucleating effect of the excess carbide on the decomposition of austenite on quenching. As was demonstrated by the authors, the nucleating effect of undissolved carbide particles is doubtlessly an important contributing factor. However, I wonder if the authors have considered the effect of variation of heat conductivity due to the change of carbon content in the given series.

When I was in Lehigh University last year, Dr. R. D. Stout and I did some work on the hardenability of steels. Some of the results may throw some light on this question.

We made the Jominy hardenability test on a S.A.E. 1035 steel, all of the specimens being machined from one heat. For special purposes, the specimens were heated to 2100 degrees Fahr. (1150 degrees Cent.), held for 4 hours, and then end-quenched. During the holding period, by some special arrange-

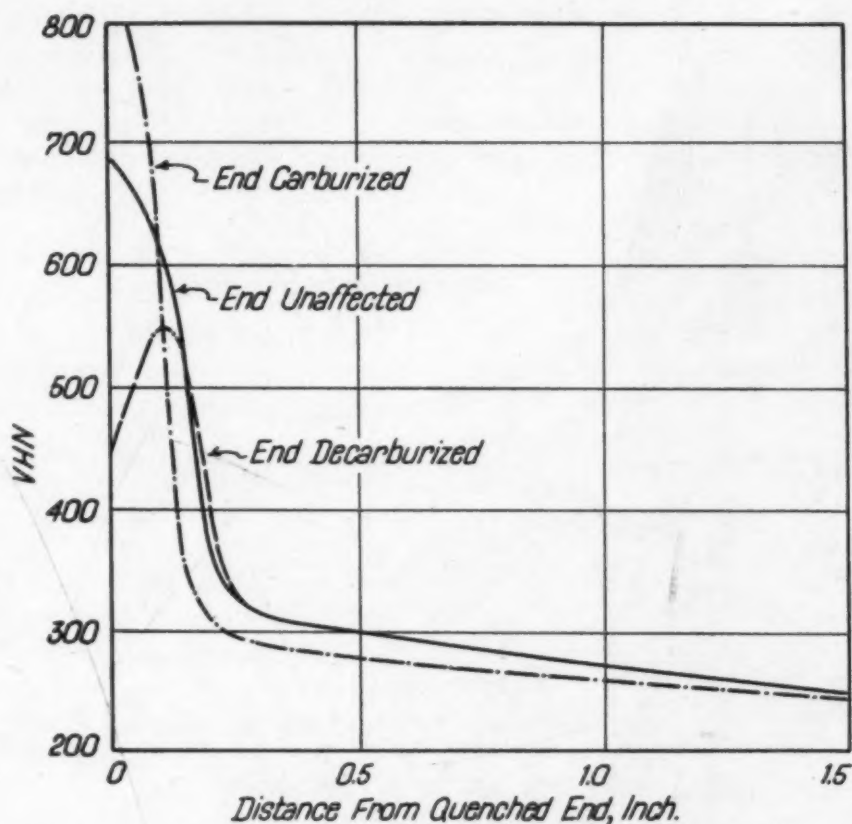


Fig. B—Hardenability Curves for a S.A.E. 1035 Steel Heated at 2100 Degrees Fahr. for 4 Hours with End Conditions as Indicated.

ments, the ends of several specimens were intentionally carburized, while those of some others were intentionally decarburized. The results are plotted in Fig. B.

After the measurements of hardness, the carburized or decarburized conditions were checked by determining the carbon content of samples from the quenched end, milled in 0.025-inch steps after the specimens were softened. Representative results are shown below:

0.025-Inch Step from Quenched End	Per Cent Carbon in a Carburized Specimen	Per Cent Carbon in a Decarburized Specimen
1	0.89	0.12
2	0.79	0.21
3	0.63	0.27
4	0.51	0.32
5	0.46	0.33
6	0.40	0.37
7	0.37	0.38

These results demonstrate clearly that the hardenability of specimens with carburized end is much lower, and that with decarburized end is slightly higher, than that of the normal, unaffected specimens. What causes this difference?

Since the specimens were held at 2100 degrees Fahr. (1150 degrees Cent.) for 4 hours, the possibility of the existence of undissolved carbide was ruled out. Furthermore, the carbon content at the transition zone (50 per cent mar-

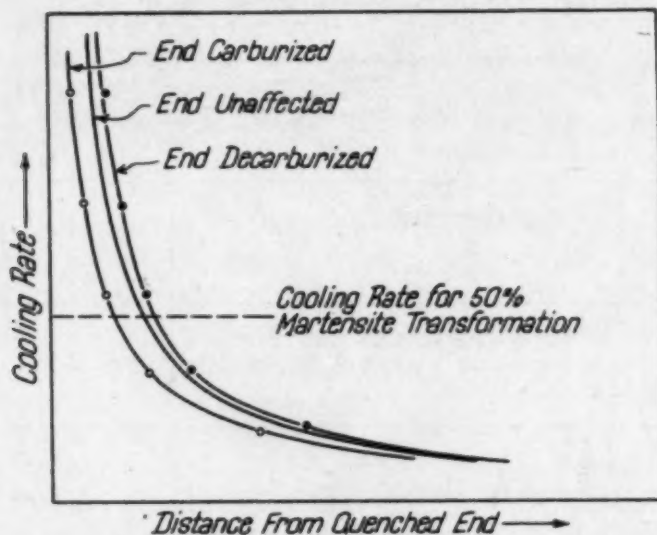


Fig. C—Cooling Rate Curves of Jominy Specimens with End Conditions as Indicated. The Jominy Distance of the Point Where the Dotted Line Intercepts the Cooling Rate Curve for a Given Carbon Content Gives the Hardenability of That Carbon Content.

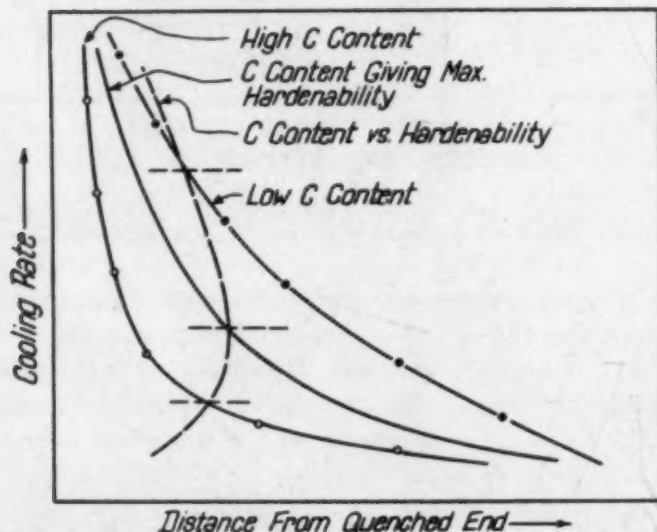


Fig. D—Cooling Rate Curves of Jominy Specimens with Various Carbon Contents. The Short Dotted Lines Show the Proper Cooling Rates for a 50 Per Cent Martensite Transformation for Various Carbon Contents. The interception points of these lines with the cooling rate curves denote the hardenability of a given series of steels having various carbon contents. Also, the hardenability curve with respect to the carbon content.

tensite zone or approximately the inflection point of the hardenability curve) was changed by neither the carburization nor the decarburization of the end. The only explanation which we can offer is that the rate of the extraction of heat from the specimens during the end quenching is influenced by the variation of heat conductivity due to the change in carbon content at the ends of the specimens. It is a fundamental property that the heat conductivity decreases as the amount of alloying element in solution (in this case carbon) increases.

If the end of a specimen is carburized, its heat conductivity is lowered, and consequently, the extraction of heat from the specimen during the quenching is retarded. This will result in a shift of the curve of cooling rate along the Jominy specimen (as the one shown on page 74 in "Hardenability of Alloy Steels" published by A.S.M.) towards the left or the quenched end. (There is also a little change in the shape of the curve which may be neglected for the present discussion.) This means a decrease in hardenability, as the transition zone has its proper cooling rate. On the other hand, if the end of a specimen is decarburized, the hardenability will increase. Shown schematically in Fig. C.

However, in the authors' experiments, if the carbon content is increased, the proper cooling rate for a 50 per cent martensite transformation, as well as the heat conductivity, is decreased. The decrease of the proper cooling rate increases the hardenability, while the decrease of the heat conductivity decreases the hardenability. These two factors act in opposite directions and will compensate each other. If the hardenability is plotted against the carbon content, there will be a maximum hardenability at that carbon content when the effects of these two factors exactly nullify each other. This is indicated in Fig. D by the dotted and dashed curve.

Thus, by this reasoning, we are able to explain the decrease of hardenability with the increase of carbon content over a certain percentage, even if all of the carbides go into solution before quenching.

Written Discussion: By O. W. McMullan, Bower Roller Bearing Company, Detroit.

The writer is greatly interested in this entire paper but wishes to comment on only one point. The authors conclude, from results on the high carbon steels in their series, that there would be considerable difficulty in obtaining reproducible results from the carburized end quench test to measure case hardenability. This difficulty arises from the variation in hardenability caused by a few points difference in carbon content.

By taking a different viewpoint this apparent disadvantage might prove to be a distinct advantage for the end quench test for this application. If the specimen were carburized so as to possess a hyper zone of considerable depth then a single specimen would represent all the possibilities as far as carbon is concerned. Since cooling rates in the end quench specimen are dependent on distance from the quenched end rather than from the surface tested, all zones in the case are quenched at the same rate, and their hardenability can be determined with one specimen by merely testing the surface and flats ground to various depths. Microscopic examination of a cross section would show the carbon content at the various levels and such a section could be taken at the cooling rate that would show the carbon content to best advantage. Results then should

be comparable from heat to heat and would at the same time determine the carbon range (particularly the maximum limit) necessary to meet the required hardness.

Written Discussion: By W. E. Jominy, chief metallurgist, Dodge Chicago Plant, Division of Chrysler Corp., Chicago.

This paper contains much useful data. It has added to our knowledge of the behavior of alloy steels in the carbon range we have with carburized parts. It points out the unexpected result that under certain conditions we may lack full hardening at the surface on carburized parts if we have absolutely no decarburization. In aircraft work we seem to have more difficulty from austenitic surfaces and are compelled in many instances to supercool to obtain the desired hardness.

In automotive applications the tendency is to direct quench without reheat and here again the difficulty seems to be too much austenite. This has resulted in the so-called hesitating quench in which the pieces are permitted to air cool to about 1350 degrees Fahr. (730 degrees Cent.) before quenching. It has been assumed that this practice permits time for carbides to precipitate and these nucleate the austenite-martensite change. It would be expected, however, that if the hardenability increases as the carbon concentration decreases from about 1.15 per cent carbon, we would have more austenite on the surface due to less carbon in solution. Perhaps the authors may have some ideas on this subject.

A conclusion which we may arrive at from the authors' data is that much higher hardenability can be obtained in carburized parts if the carbon concentration at the surface can be kept at the low value of about 0.70 per cent. It is generally conceded, however, that high carbon gives better wear resistance than lower carbon so that for wearing parts this procedure may not be desirable.

Written Discussion: By T. L. Counihan, chief metallurgist, Hyatt Bearings Division, General Motors Corp., Harrison, N. J.

The authors have given us evidence as to the influence of carbon concentration on hardenability. They amply demonstrate its potency.

In general, the prime purpose of most alloying elements is to increase hardenability. The fact that too high a carbon content can reduce hardenability is oftentimes overlooked or at least is not given proper consideration.

The present speaker can add little to the authors' writing except to substantiate their results.

Had the speaker any thought that the data he is to offer were to serve a purpose here today, he would likely have used a specimen much like the end-quenched Jominy bar, rather than the cylindrical bar, which actually was a roller for a bearing and used because it was, at the time, the object of an investigation. With the audience understanding this point, he would like to say that the two charts presently to be shown depict the influence of carbon concentration on the hardenability of a S.A.E. 4320 steel of the following composition:

Per Cent					
Carbon	Manganese	Silicon	Nickel	Chromium	Molybdenum
0.20	0.57	0.23	1.78	0.48	0.25

Fig. E illustrates the influence of carbon concentration on hardness as related to a variable cooling rate.

The solid cylindrical specimen was 3.50 inches in length and 1.68 inches in diameter. It was carburized at 1700 degrees Fahr. (925 degrees Cent.) to a depth of 0.150 inch, quenched in oil, reheated to 1510 degrees Fahr. (820 degrees Cent.), and quenched in water to a depth of 1.25 inches. This location is noted by the vertical line AB between the numerals 9 and 11 at the bottom of Fig. E where a sketch of the specimen is located. The portion of the specimen to the right of the vertical line is cooled in air at a variable rate, depending upon its distance from the water line.

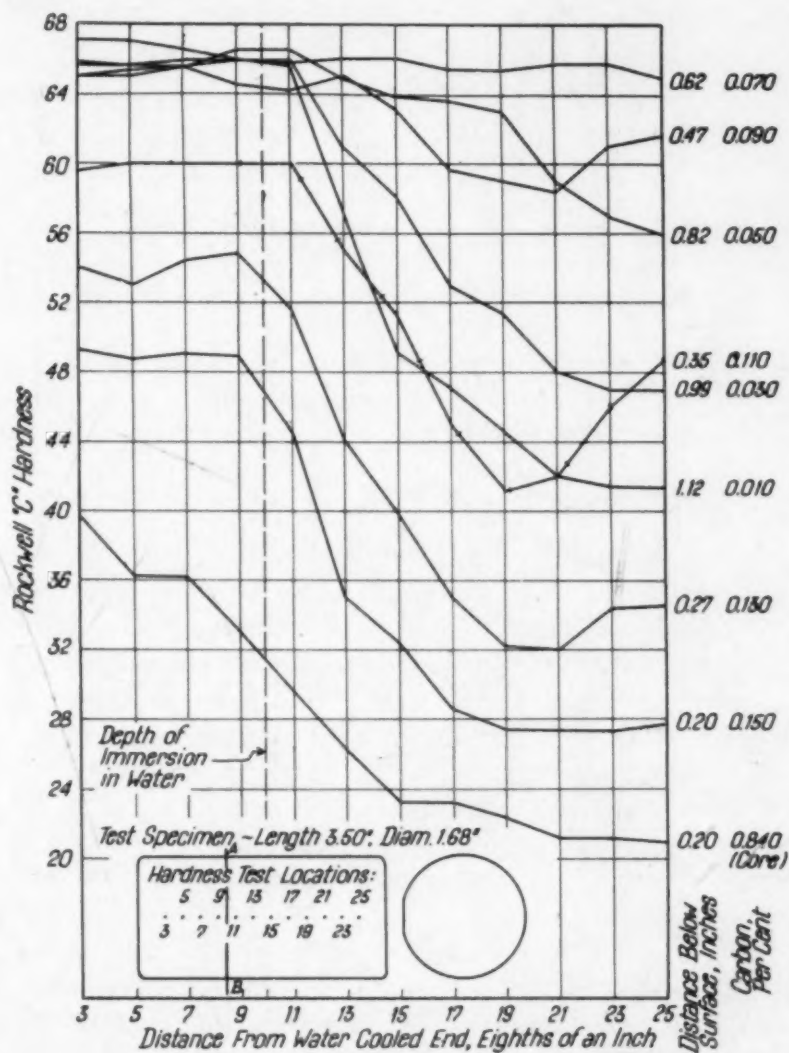


Fig. E

Flats were ground on the bar in 0.010-inch increments and hardness readings were made at distances $\frac{1}{8}$ inch apart along its length. To avoid causing confusion by plotting a large number of curves, although readings on some forty flats were made, only nine representative curves are shown. At corresponding increments carbon analyses were made on three other cylinders carburized at the same time so that it is possible to state what the carbon concentrations were at these varying levels. These carbon concentrations are shown at the right of the respective curves.

One may note that as the carbon concentration, which was 1.12 per cent at a depth of 0.010 inch beneath the surface, decreases to 0.99 to 0.82 and 0.62 per cent, the hardenability increases. At about 0.62 per cent carbon maximum hardenability is reached. Thenceforth the hardenability decreases as the carbon drops to 0.47 to 0.35 to 0.27 per cent and finally to the core concentration of 0.20 per cent, where the lowest hardenability is found. It is interesting to

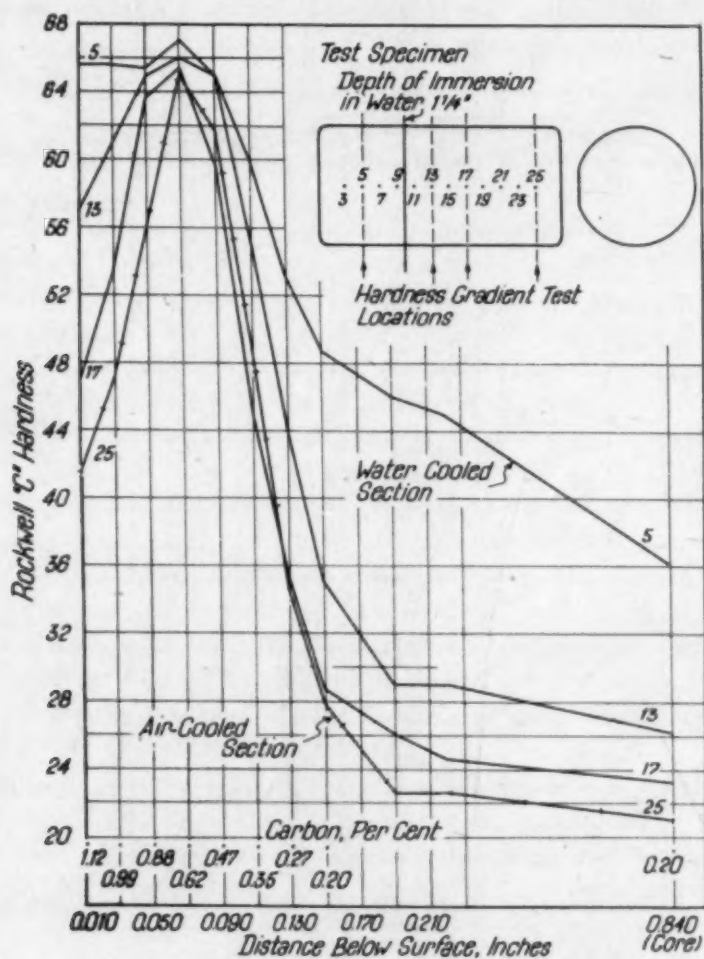


Fig. F

note that the hardness of 1.12 per cent carbon nearly corresponds to that of a 0.35 per cent carbon steel except where the cooling rate is rapid. Also, that the 0.62 per cent carbon curve assumes practically a straight line, indicating considerable hardenability. Fig. F is merely another way of presenting the information shown in Fig. E. Four curves are shown illustrating the fact that irrespective of cooling rate maximum hardenability occurs at about 0.62 per cent carbon.

The curve bearing the notation 25, which indicates it represents the cross sectional hardness at a distance 25/8 or 3 1/8 inches from the water-quenched end and hence has cooled relatively slowly, shows the potency of carbon concentration. At 0.010 inch beneath the surface, where the carbon concentration is 1.12 per cent, the hardness obtained is only Rockwell C-41.5, while at a depth

of 0.070 inch beneath the surface, where the carbon concentration is but 0.62 per cent, the hardness is Rockwell C-65.

In closing, the speaker wishes to say that he feels the authors' results indicate the desirability of controlling the carbon concentration on carburized parts. An unduly high carbon concentration can result in an extravagant apparent need for higher alloy than necessary to obtain satisfactory hardness values.

Authors' Closure

Before considering the discussions offered on this paper, the authors would like to add a small amount of additional data obtained since the paper was written. The 46XX series suffered somewhat from the absence of data at the higher carbon contents often encountered in carburizing—i.e., about 1.20 per cent carbon. Consequently, two additional induction heats of the following composition were melted and subjected to exactly the same procedure as the foregoing heats.

Heat	C	Mn	P	S	Si	Cr	Ni	Mo	Brinell Spheroidized	Hardness Normalized
1480	1.22	0.45	0.016	0.035	0.30	0.25	1.66	0.28	217	363
1481	1.27	0.45	0.016	0.035	0.32	0.24	1.68	0.28	217	363

Fracture grain size values obtained under the conditions of test are listed below:

Heat	Per Cent Carbon	Time at Temperature (1550 Degrees Fahr.)							
		0 Min.		10 Min.		40 Min.		4 Hours	
		Ann.	Norm.	Ann.	Norm.	Ann.	Norm.	Ann.	Norm.
1480	1.22	8	8	8	8	8	8	7½	8
		40 Min. at Quenching Temperature, Degrees Fahr.							
		1450		1500		1550		1600	
		Ann.	Norm.	Ann.	Norm.	Ann.	Norm.	Ann.	Norm.
1481	1.27	8	9	7½	8½	7	8	5½	7½
									6

The results of the hardenability tests are incorporated in Figs. G and H which may be substituted for Figs. 9 and 10 of the text. The trend is certainly as expected and, in particular, shows continued general decrease in hardenability with this increase in carbon content. Furthermore, continued convergence of the hardenability values at higher carbon contents with quenching temperature variation is definitely indicated.

In regard to Mr. Digges' discussion of the paper, we certainly owe an apology for overlooking the reference he cites. Unfortunately, the Journal was not immediately available and the abstract we did observe gave no hint of additional work beyond that contained in reference 3. Nevertheless, we are happy that our results on alloy steels so completely conform with Mr. Digges' work on carbon steels. This certainly serves as further confirmation of the general relationship between carbon content and hardenability.

The authors are in complete agreement with Mr. Digges' statements in regard to the difficulty in determining the austenitic grain size of hypereutectoid steels. The fracture method, while empirical, is generally conceded to be a reliable relative measure of austenitic grain size and correlations with A.S.T.M. grain size have been made. Since carbon solution and grain growth in these steels are apparently so completely interdependent, the authors are unable to

answer the question whether the presence of spheroidized carbides alters the appearance of the fracture. If one accepts the plausible theory that austenitic grain growth in hypereutectoid steels is a result of carbide solution and the fact that the microscopic delineation of grain size in these steels is uncertain, the fracture method automatically becomes the most likely means of estimating grain size.

Mr. Jominy has raised the question of high austenite contents in the microstructure at the surface of carburized parts and how such a condition corre-

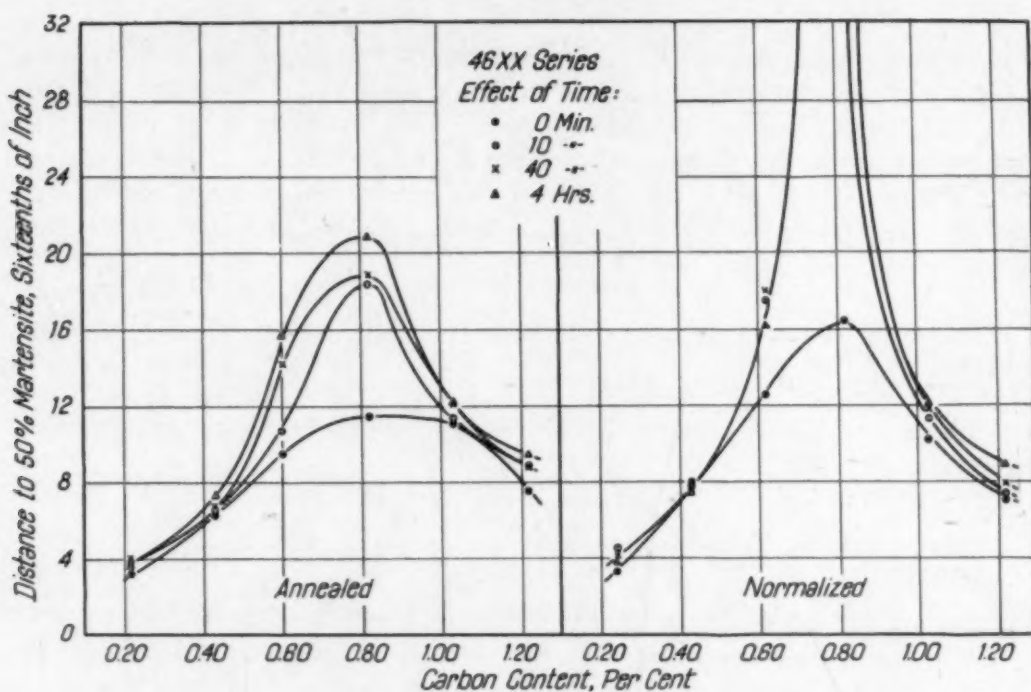


Fig. G—Effect of Carbon Content on Hardenability of the 46XX Series as Influenced by Time at 1550 Degrees Fahr. (Extension of Fig. 9.)

lates with the hardenability-carbon content relationship. The belief is held in some quarters that the beginning of the A_r'' transformation, and probably its ending as well (the M_s and M_f points), are not time-sensitive functions and hence the austenite to martensite transformation is not nucleated by either carbide or ferrite but, in a given steel, is dependent solely upon the amount of carbon in solution at the moment of quenching. The A_r'' range is progressively and continuously lowered as the amount of carbon in solution increases and the amount of retained austenite in the quenched structure increases accordingly. The austenite to quenching pearlite transformation, on the other hand, is apparently nucleated by excess carbide and the hardenability as measured to a given amount of decomposition product on the bar decreases rapidly with the appearance of excess carbide. These considerations lead to the conclusion that, at a given quenching temperature and under substantially equilibrium conditions the tendency to retain austenite on quenching increases with carbon content up to the limit of solubility and remains essentially constant with further increase in carbon while the hardenability decreases very rapidly beyond the solubility limit. In commercial heat treatment where equilibrium is by no means ap-

proached, the expected faster rate of carbon solution with increasing carbon content in the hypereutectoid range would apparently allow the retained austenite to increase in amount while the hardenability was decreasing. Fig. 4d of the paper shows that the quenched end of the 0.79 per cent carbon bar is almost completely martensitic while the 1.03 per cent carbon bar contains substantial quantities of austenite. The ultimate conclusion reached with this line of reasoning is that the austenite content of quenched pieces can be controlled at small values by reducing the amount of carbon in solution at the moment of

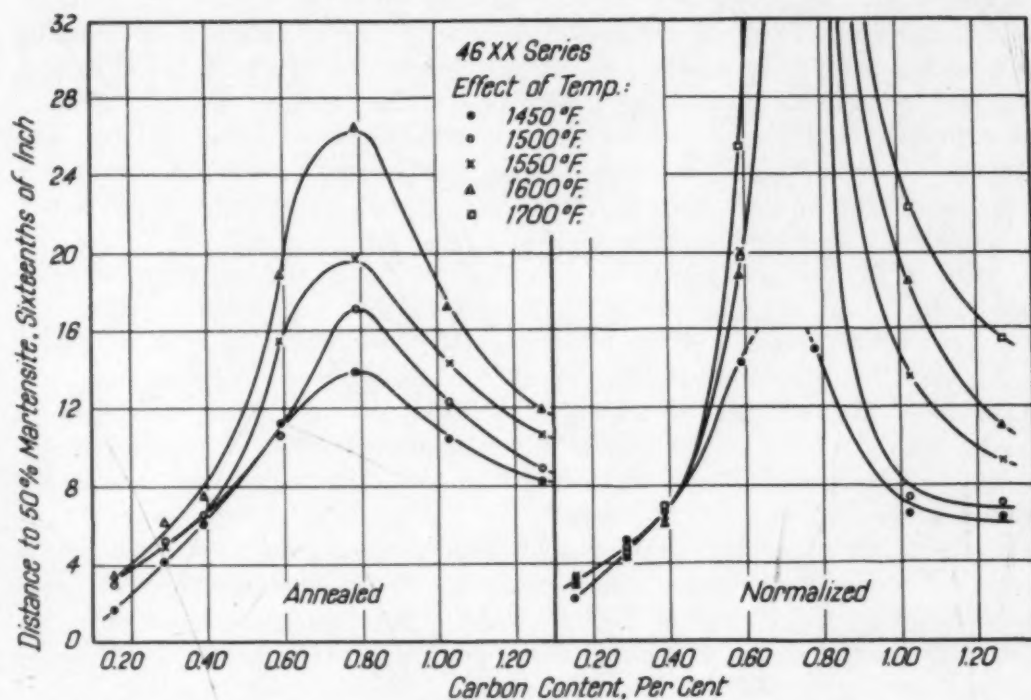


Fig. H—Effect of Carbon Content on Hardenability of the 46XX Series as Influenced by Quenching Temperature (40 Minutes at Temperature). Extension of Fig. 10.

quenching, either by reducing the maximum carbon concentration in carburizing or by interrupting the quench to allow some carbide to precipitate as cited by Mr. Jominy. A further example is the tempering of carburized and pot quenched S.A.E. 3312 to precipitate excess carbide which does not readily dissolve on reheating and thus minimizes austenite formation on hardening.

The authors certainly did not wish to recommend maximum carbon concentrations for carburizing in the range of 0.70 per cent carbon since the advantages of excess carbide in the case structure are well known. The authors know of situations, however, where soft spots on the surface of pack carburized and pot-quenched articles were blamed on decarburization whereas the maximum carbon concentration was actually high enough to produce soft spots through low hardenability. Our own experience with S.A.E. 4620 steel leads us to believe that a maximum carbon concentration of about one per cent is an effective compromise between the opposing considerations of wear resistance and case hardenability.

The discussion offered by Mr. Sun was studied with great interest since the effect of change in thermal conductivity was not considered in our interpre-

tation of the data contained in this paper. Mr. Sun has shown that an increase in carbon content will decrease the thermal conductivity of the bar and hence tend to decrease the hardenability measurement but he has not shown that the maximum will occur at a carbon content compatible with the experimental evidence. He also has not shown why the decrease in hardenability beyond the carbon solubility limit is so rapid when, by his interpretation, one would judge that the decrease would be much more gradual. In the absence of more complete evidence, the authors prefer to believe that the effects of carbon in solution and as excess carbides control the hardenability variation and that the influence of thermal conductivity is of minor importance. For strict scientific accuracy, the cooling rates along the bar should be corrected for the effect of change in thermal conductivity just as they should be corrected for the effect of variation of quenching temperature. In the ultimate practical sense, of course, these corrections are unimportant since the thermal conductivity of the end quench bar and the production part from the same steel are the same and the production quenching temperature should be used in performing the test.

Mr. McMullan's viewpoint in regard to the carburized end quench test is most interesting and, if all the precautions outlined by him could be taken, the test can be interpreted with entirely satisfactory accuracy. It must be recognized, however, that microscopic estimation of the carbon content at any cooling rate on the bar offers some important difficulties particularly in the range where 0.05 per cent carbon means a hardenability variation of several sixteenths. Even chemical analysis for carbon content is a problem because the carbon gradient is changed in softening for machining and the absence of centers makes accurate machining of small cuts something of a problem. This point was raised in the paper only because it was thought that the very great influence of a small change in carbon content in the case hardenability was not generally recognized and that many of the disagreements in hardenability measurement by this method were due to the lack of adequate control of this factor.

Mr. Counihan very ably demonstrates in one sample the entire effect of carbon content on hardenability and is to be congratulated on the ingenuity of the means used. The discrepancy in regard to the carbon content at maximum hardenability between his results and ours may be due to a distortion of the cooling rate at some levels caused by quenching part of the specimen from both the end and the cylindrical surface.

The authors wish to thank the discussers of this paper for the time and effort taken in these busy times to prepare these discussions which have added so materially to the value of this paper.

AIR HARDENABILITY OF STEELS

BY C. B. POST, M. C. FETZER AND W. H. FENSTERMACHER

Abstract

The center cooling velocity of regular shapes during air hardening is shown to be controlled by the area per unit volume (A/V) relationship and is independent of treating temperature.

A gradient air hardenability test is described. This test enables a determination of the air hardening properties of a given air hardening steel to be made ranging from the equivalent of a 1-inch round by 8-inch long cylinder to a 6-inch round by 6-inch long cylinder. Air hardenability is stated here in terms of the cooling velocity with which the steel must be cooled past 1000 degrees Fahr. (540 degrees Cent.) to obtain a given R_c hardness.

The effects of treating temperature, soaking times and rate of heating through the critical on the resulting hardenability of several air hardening steels are described.

In most cases it is possible to make a good approximation of the A/V value of the tools to be heat treated. Combining this value with the hardenability of the steel to be used, it is a simple matter to estimate with accuracy the hardness to be obtained throughout the tools by air treatment.

The hardenability of several air hardening steels is presented.

In general the variation in air hardenability of air hardening steels is negligible from heat to heat for a given type analysis.

AIR hardening steels have definite advantages in certain types of tool steel applications due to the ease of heat treatment, relatively small distortion after hardening, and, probably most important, the absence of appreciable thermal gradients during quenching, resulting in practically complete freedom of quench-cracks in intricate sections. In contrast to the abundance of hardenability data relating to water and oil hardening types of steel, practically no

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quantitative data on the hardenability of air hardening types of steel are available.

It is the purpose of this paper to describe an air hardenability test and a method of stating air hardenability in such a manner that these data can be used by tool makers and heat treaters to aid in the selection of air hardening steels. The test developed consists of air-cooling a 1-inch round bar in such a manner that one end is allowed to cool freely, while the cooling of the other end is retarded to a rate equivalent to that of a 6-inch round by 6-inch long cylinder. Cooling velocities through a temperature range are known along the length of the 1-inch round bar. A determination of the hardness gradient along the 1-inch round bar enables the Rockwell hardness to be stated as a function of these cooling velocities. The parameter found to give the closest agreement between hardness predicted from the air hardenability test and that obtained by actual heat treatment of various size sections is the cooling rate (degrees Fahr. per minute) past 1000 degrees Fahr. (540 degrees Cent.).

The most obvious application of the above outlined hardenability test is the prediction of the hardness to be expected of a given air hardening steel when air-treated in various sized pieces. The application of these data is simplified by the fact that cooling rates in the vicinity of 1000 degrees Fahr. (540 degrees Cent.) are equal throughout the mass during air hardening, except for extremely irregular shapes having fins, pins, etc., protruding from the larger mass. This means that air hardening leads to the same hardness throughout the mass from center to surface. Thus, knowing the center cooling rate (degrees Fahr. per minute past 1000 degrees Fahr.) of a regularly shaped mass, and the air hardenability of a given air hardening steel (R_c hardness as a function of degrees Fahr. per minute past 1000 degrees Fahr.), it is easy to predict the hardness obtained in the mass when using this air hardening steel.

The simplicity of this application of air hardenability data depends to a large measure on the effect of (a) shape of piece (round, rectangular, long or short), and (b) treating temperature on the cooling rate of a regularly shaped mass of steel. In Part I it is shown that the air-cooling rate (past 1000 degrees Fahr.) of regularly shaped masses is a function only of the surface to volume ratio, and further, this cooling rate (past 1000 degrees Fahr.) for a given surface to volume ratio appears to be independent of treating temperature.

In Part II an air hardenability test is described. Air-cooling rates past 1000 degrees Fahr. (540 degrees Cent.), ranging from those of a 1-inch round by 8-inch long (Surface/Volume = 4.25) to a 6-inch round by 6-inch long specimen (Surface/Volume = 1) are obtained simultaneously by means of this method. This permits the hardness developed by an air hardening steel to be determined as a function of the cooling rate past 1000 degrees Fahr. (540 degrees Cent.).

In Part III the air hardenability of various well-known types of air hardening steels is presented. The influence of several heat treating variables, such as treating temperature, soaking time at treating temperature, and rate of heating through the critical range on the resulting air hardenability, is studied. It is shown that the treating temperature and soaking time, as expected, affect the resulting air hardenability, but the rate of heating through the critical appears to have no effect.

PART I

AIR-COOLING CHARACTERISTICS OF REGULARLY SHAPED MASSES

1. *Cooling Rates of Rounds*—Fig. 1 shows the center and surface cooling curves of a 6-inch round by 6-inch long specimen when air-treated from 1600 degrees Fahr. (870 degrees Cent.). 30 per cent nickel steel was used because of its known similarity in thermal diffusivity to that of austenite. The surface thermocouple was $\frac{1}{8}$ inch below the surface of the round. Although the surface temperature drops rapidly to about 160 degrees Fahr. below that of the center when the specimen is first brought into the air, an analysis of the two curves shown in Fig. 1 leads to the conclusion that the rates of cooling of surface and center are practically equal, especially in the temperature ranges of 1300, 1000 and 600 degrees Fahr. (705, 540, and 315 degrees Cent.). This indicates that the hardness across the section of a regularly shaped mass should be constant from surface to center. This has been verified repeatedly by hardness contours on the cross section of various sized regular shapes made from air hardening steels. As a matter of fact, actual surface hardnesses have been found to be equal to the hardness throughout the section when the steels are properly heat treated to avoid decarburization.

The influence of diameter and length of round cylinders on the

center cooling rate past several temperatures during air hardening was next investigated. For these experiments 32 per cent nickel steel was used, together with a constant initial temperature of 1600 degrees Fahr. (870 degrees Cent.). Fig. 2 shows the experimental methods used for determining these center cooling rates of

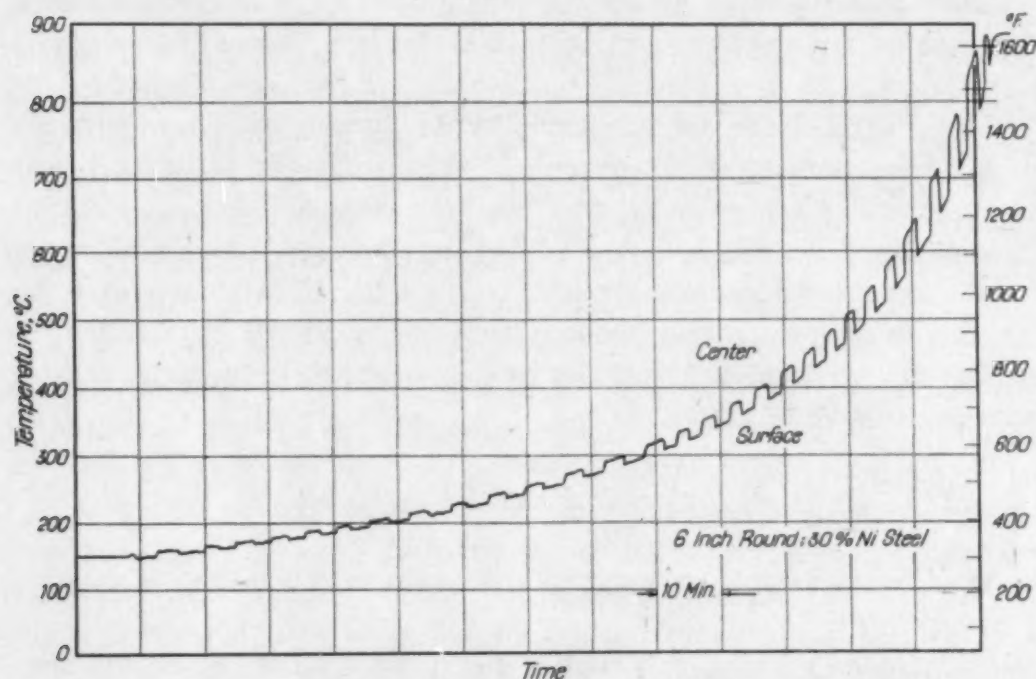


Fig. 1—Center and Surface Air-Cooling Curves of a 6-Inch Round by 6-Inch Long Cylinder of 32 Per Cent Nickel Steel.

various sized rounds. For rounds 3 inches or more in diameter, the method shown on the left was used. For sizes of rounds 2 inches in diameter and smaller, the method shown on the right was used.

Center cooling rates (degrees Fahr. per minute) past 1000 degrees Fahr. (540 degrees Cent.) and past 1300 degrees Fahr. (705 degrees Cent.) for these various sized rounds are given in Table I. The 1000-degree Fahr. (540-degree Cent.) cooling rate represents the average from 1075 to 925 degrees Fahr. (580 to 495 degrees Cent.), and the 1300-degree Fahr. (705-degree Cent.) cooling rate represents the average from 1375 to 1225 degrees Fahr. (745 to 665 degrees Cent.). When these cooling rates are plotted against A/V , where A is the area of the cylinder, and V is the volume, a linear relationship results for each set of cooling rates, i.e., past 1000 degrees Fahr. (540 degrees Cent.) and past 1300 degrees Fahr. (705 degrees Cent.).

Table I
Experimental Cooling Rates for Rounds and Rectangular Shapes

Size	A/V	In. of Corner Per Unit Volume	Cooled from 1600° F.			Cooled from 1900° F.		
			Rate Past			Rate Past		
			1000°	1300°	1300° 1000°	1000°	1300°	1300° 1000°
32 Per Cent Nickel Steel								
1" dia. x 1"	6.0	8.0	163	288	1.77
1" dia. x 1"	6.0		166	298				
1" dia. x 2"	5.0		131	240				
1" dia. x 2"	5.0	4.0	131	240	1.83
1" dia. x 3"	4.67		121	211				
1" dia. x 3"	4.67	2.67	121	211	1.83
1" dia. x 4"	4.5		115	109	200	1.83
1" dia. x 4"	4.5	2.0	115	206	1.79			
1" dia. x 6"	4.33		108	197				
1" dia. x 6"	4.33	1.33	108	200	1.83
1" dia. x 8"	4.25		89.5	173	1.93
1" dia. x 8"	4.25	1.0	101	184	1.82
2" dia. x 2"	3.0		75.8	135				
2" dia. x 2"	3.0	2.0	75.2	140	1.82
2" dia. x 3"	2.67		63.5	115				
2" dia. x 3"	2.67	1.33	65.5	120	1.82
2" dia. x 4"	2.50		60.5	108				
2" dia. x 4"	2.50	1.0	60.0	111	1.82
2" dia. x 6"	2.33		55.3	98				
2" dia. x 6"	2.33	0.67	55.4	98	1.77
2" dia. x 8"	2.25		52.5	92	1.75
2" dia. x 8"	2.25	0.50	51.2	92	1.79
3" dia. x 3"	2.00		46.6	85.6		48	90	1.87
3" dia. x 3"	2.00	0.88	44.6	80.0	1.82	43.6	80	1.83
4" dia. x 4"	1.5		31.5	56.5				
4" dia. x 4"	1.5	0.50	31.8	57.7	1.80
6" dia. x 6"	1.0		21.0	38.5	1.83	21.4	39.0	1.82
6" dia. x 6"	1.0	0.22	23.7	43.6	1.84
5.44 x 5.44 x 5.44"	1.1	0.41	21.6	39.5	1.83	20.8	38.9	1.87
5.44 x 5.44 x 1.63"	1.955	1.04	41.5	76.2	1.83	42.9	74.0	1.73
5.44 x 1.63 x 1.50"	2.932	2.58	67.5	113.5	1.68	69.5	125	1.80
5.44 x 1.63 x 1.50"	2.932	2.58	67.5	123	1.82
1.50 x 1.50 x 1.63"	3.9	5.05	90.5	176	93	174	1.87
1.50 x 1.50 x 1.63"	3.9	5.05	90.0	173	1.92
1.50 x 1.50 x 1.63"	3.9	5.05	90.5	173
Air Hardening Steels								
Steel A*								
2.75 x 2.75" rd.	2.18	1.04	45.2	86.5	1.91	85.6
Steel B*								
1.75 x 1.75" rd.	3.44	2.62	77.0	146.2	1.90	76.5	144	1.88
Steel T*								
6" dia. x 6"	1.0	0.22	19.9	35.6	1.79

*Refer to Table IV for Analyses of Steels Used.

When plotted logarithmically, these two straight lines become parallel as shown in Fig. 3. The above data appear as open circles in Fig. 3. The A/V range represented is from 1 to 6 (6 inches round by 6 inches long to 1 inch round by 1 inch long).

The A/V for the 1-inch rounds investigated ranges from 4.25 to 6.0 (8 inches long to 1 inch long) and all of the open circles in Fig. 3 in this range represent 1-inch diameter rounds. It is to be noted that this family of points departs more and more from the straight line as A/V increases. This is due to corner effect. The linear

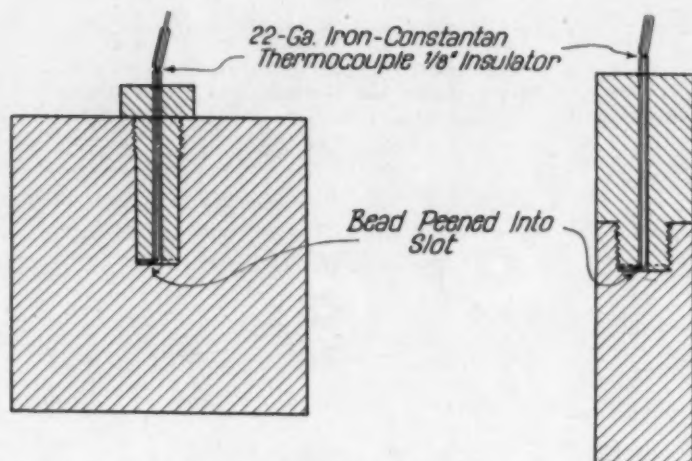


Fig. 2 (Left)—Experimental Technique for Rounds 3 Inches in Diameter or Larger. (Right)—Experimental Technique for Rounds 2 Inches in Diameter or Smaller.

length of corner per unit volume of metal increases as follows for 1-inch rounds of decreasing lengths:

Length Inches	A/V	Inches of Corner/Volume
8	4.25	1.0
6	4.33	1.3
4	4.50	2.0
3	4.67	2.7
2	5.00	4.0
1	6.00	8.0

A similar effect is noted with the 2-inch rounds represented by the open circles between $A/V = 2.25$ (8 inches long) and 3.00 (2 inches long). Here the inches of corner per unit volume range from 0.50 inch (8 inches long) to 2.00 inch (2 inches long).

The curves of Fig. 3 were drawn to agree with low values of inches of corner per unit volume.

2. *Cooling Rates of Bars, Slabs, and Blocks*—Cooling rates were next measured on other shapes to determine the relationship between the center cooling rates and the A/V ratios. The procedure consisted of taking a 6-inch cube of 32 per cent nickel steel, determining the center cooling rate from an initial temperature of 1600 degrees Fahr. (870 degrees Cent.), and then decreasing the dimensions, changing the shape, and increasing the A/V according to the following schedule:

Size, Inches	Shape	A/V
6 x 6 x 6	Large Cube	1
6 x 6 x 1.5	Slab	2
6 x 1.5 x 1.5	Column	3
1.5 x 1.5 x 1.5	Small Cube	4

The center cooling rates and actual size of the pieces are given

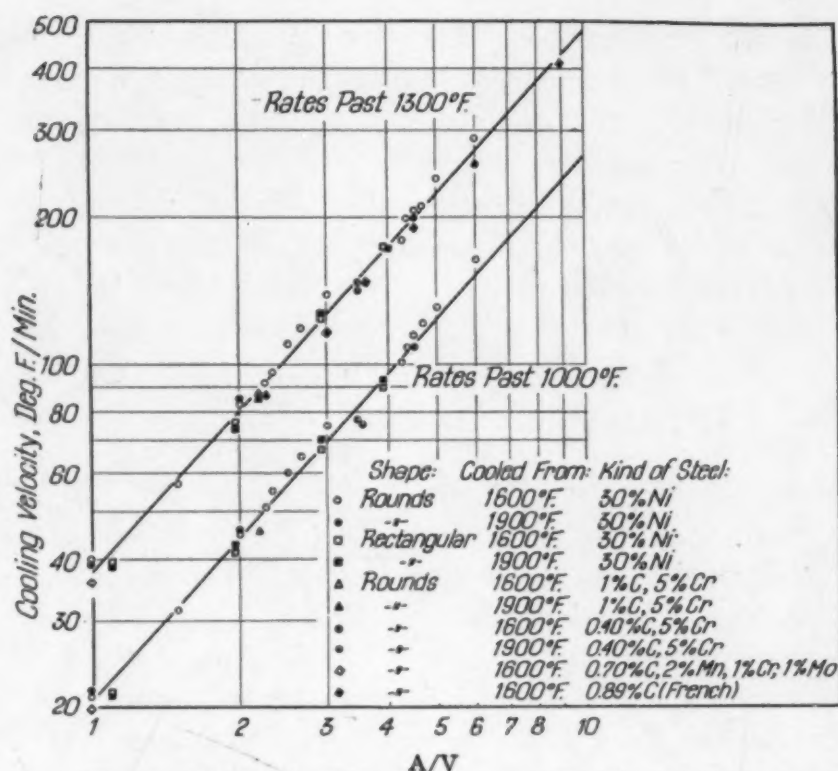


Fig. 3—Center Air-Cooling Rates as a Function of the Area to Volume (A/V) Ratio for Regularly Shaped Masses.

in Table I and plotted as open squares in Fig. 3. The cooling rates of these shapes are the same as rounds having the same A/V values.

3. *The Effect of Austenite Composition on Cooling Rate*—All work described so far was carried out on 32 per cent nickel-iron alloy. This alloy was used because it is austenitic (face-centered lattice) at all temperatures, does not crack on repeated heating and cooling, and resists scaling. To determine whether the austenite of typical air hardening steels cools at the same rate as the 32 per cent nickel austenite, center cooling rates were determined on Steels A, B and T (see Table IV for analyses of steels), using initial temperatures of 1600 and 1900 degrees Fahr. (870 and 1040 degrees Cent.). The sizes investigated were 6-inch diameter by 6 inches (Steel T); 2.75-inch diameter by 2.75 inches (Steel A) and 1.75-inch diameter by 1.75 inches (Steel B).

These data are shown in Fig. 3 and they agree well with those obtained on the 32 per cent nickel-iron alloy. Included in Fig. 3 are some rates past 1300 degrees Fahr. from an initial temperature of 1600 degrees Fahr. (870 degrees Cent.) obtained by French¹ on

¹H. J. French, "The Quenching of Steels," American Society for Steel Treating (1930), p. 35.

an 0.89 per cent carbon steel. French's values of A/V extended up to 9 and are all in good agreement with the curve of Fig. 3.

4. *Effect of Treating Temperature*—The cooling rates described so far were mostly obtained by using an initial temperature of 1600 degrees Fahr. (870 degrees Cent.). To determine whether the initial temperature had any effect on the rates, some of the 32 per cent nickel steel test pieces were heated to 1900 degrees Fahr. (1040 degrees Cent.) and cooled. The center cooling rates obtained are given in Table I and Fig. 3. In Fig. 3 the center cooling rates from 1900 degrees Fahr. (1040 degrees Cent.) are shown as solid circles, squares, etc., corresponding to the open circles, squares, etc., which refer to cooling rates from 1600 degrees Fahr. (870 degrees Cent.). Within experimental error, the initial treating temperature has no effect on the rate of cooling through 1000 and 1300 degrees Fahr. (540 and 705 degrees Cent.).

5. *Ratio of Cooling Rates at 1300 Degrees Fahr. to That at 1000 Degrees Fahr.*—In Table I the ratio of the center cooling rate past 1300 degrees Fahr. (705 degrees Cent.) to that at 1000 degrees Fahr. (540 degrees Cent.) is computed and is practically 1.82 regardless of size, shape, or initial heating temperature. The significance of this fact is that the parameter used for air hardenability, such as the rate of cooling past 1000, or 600, or 1300 degrees Fahr., will be the same for rounds, slabs, blocks, and other regular shapes.

PART II

DEVELOPMENT OF AN AIR HARDENABILITY TEST

The test developed for this purpose consists of taking a 6-inch round by 6-inch long slug of 32 per cent nickel-iron alloy, drilling a 0.858-inch diameter hole through its center, and tapping about 1 inch at each end. (See Fig. 4). The hardenability specimen itself (Fig. 4) is 7 inches long, of which 4 inches of this length (1-inch diameter) extends from the end of the 6-inch slug and the remaining 3 inches (0.856 inch in diameter) reaches into the center of the slug. Two specimens are inserted, one into each end of the 6-inch round, the assembly is then heated to the hardening temperature, soaked at heat, removed from the furnace, placed on an elevated wire screen, and air-cooled. The specimens are then unscrewed, flats ground on two opposite sides, and hardnesses taken at locations corresponding to definite cooling rates past 1000 degrees Fahr. (540 degrees Cent.).

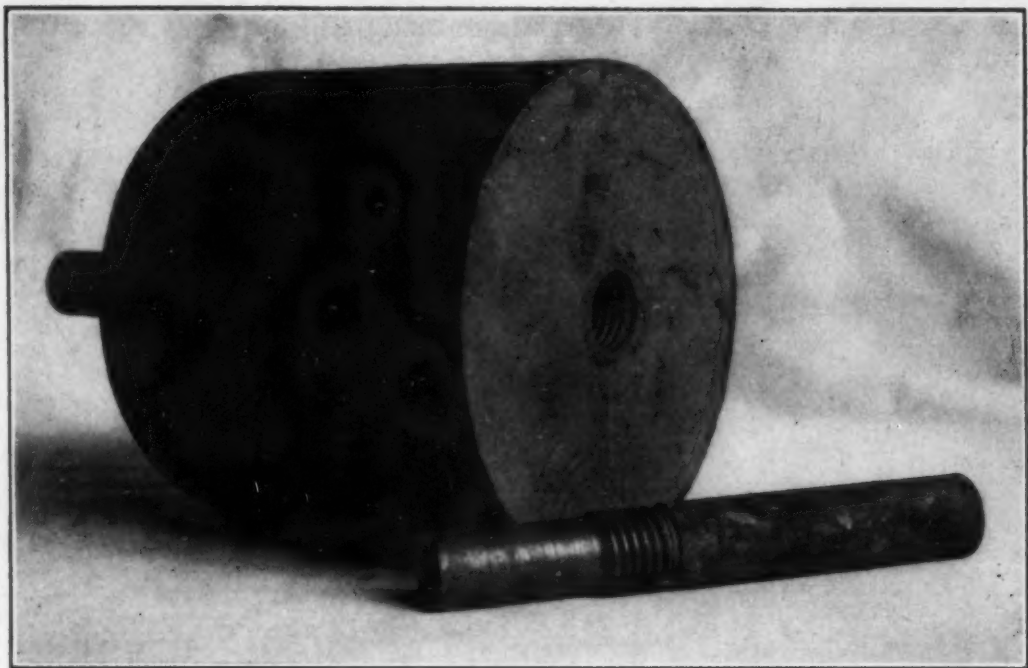


Fig. 4—Gradient Cooling Air Hardenability Test Specimen.

Tests were run to determine the cooling rate of the hardenability specimen at distances of $\frac{1}{8}$, $\frac{5}{8}$, $1\frac{1}{4}$, 2, $2\frac{3}{4}$ and $3\frac{11}{16}$ inches from the face of the 6-inch slug; at 1 inch below the face; and at the center of the 6-inch slug with the specimen in place. For these measurements $\frac{1}{8}$ -inch diameter holes were drilled to the center of the 1-inch round specimen, and a 22-gage iron-constantan couple with a 0.120-inch diameter two-hole insulator was then wedged into the hole. The assembly was heated to temperature, removed from the furnace, and air-cooled by placing on a screen. Data for time-temperature curves were obtained with a potentiometer.

Fig. 5 shows the rates past 1000 and 1300 degrees Fahr. (1075 to 925 and 1375 to 1225 degrees Fahr.). Three curves are shown on each set, one representing rates for an initial temperature of 1600 degrees Fahr. (870 degrees Cent.), one for 1750 degrees Fahr. (955 degrees Cent.) and one for 1900 degrees Fahr. (1040 degrees Cent.). Unlike the simple shapes, the cooling rate of the hardenability specimen is influenced by the initial temperature due to the feeding action of the 6-inch round slug. The higher the initial temperature, the greater this feeding action.

With this test a range of A/V from 1.0 to 4.25 is covered in one cooling cycle, i. e., 21 to 105 degrees Fahr. per minute past 1000

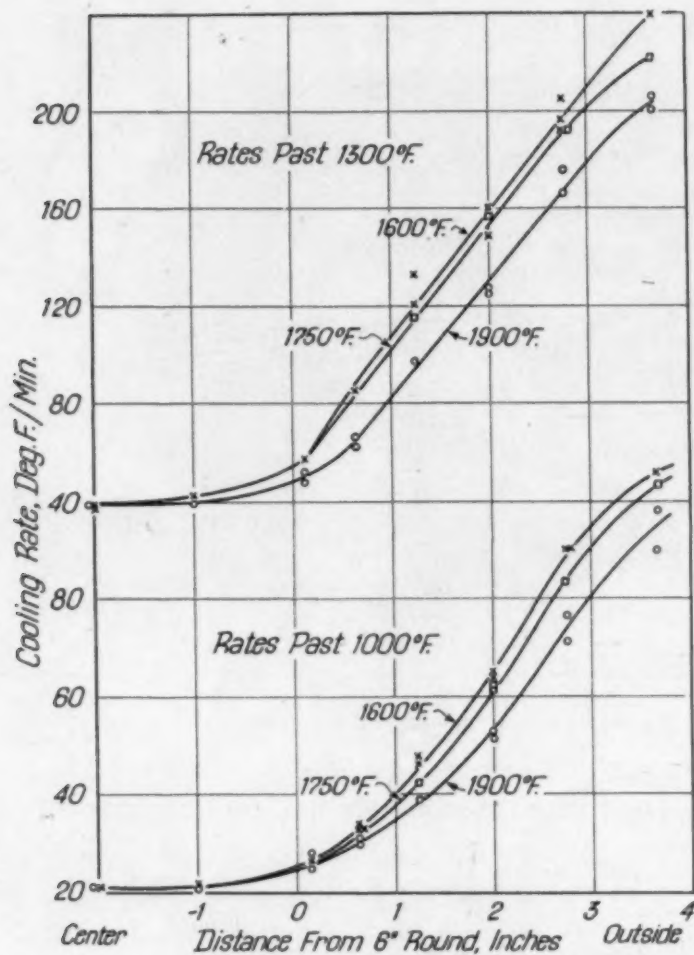


Fig. 5—Cooling Rates Along the Hardenability Specimen for Treating Temperatures of 1600, 1750 and 1900 Degrees Fahr. (870, 955 and 1040 Degrees Cent.).

degrees Fahr. A cooling rate of 124 degrees Fahr. per minute may be obtained by air-treating a 1-inch round by 2-inch long ($A/V = 5$) specimen. After heat treatment, the hardenability test specimen can be nicked and broken for fracture grain size.

It was found that the cooling curve at any given location on the hardenability specimen did not follow that of simple shapes, but instead, the cooling rate became less, relative to the simple shape, as the temperature dropped. For example, at a distance of 2 inches from the face of the 6-inch slug the hardenability specimen cools

- like a 1.62-inch diameter by 1.62-inch long piece past 1300 degrees Fahr.,
(705 degrees Cent.)
- like a 2.14-inch diameter by 2.14-inch long piece past 1000 degrees Fahr.,
(540 degrees Cent.)
- like a 3.00-inch diameter by 3.00-inch long piece past 600 degrees Fahr.,
(315 degrees Cent.)

when the initial heating temperature is 1600 degrees Fahr. (870 degrees Cent.). It therefore becomes necessary that a parameter for hardenability be chosen. Various hardenability parameters have been selected by various investigators, such as

- (a) rate past 1300 degrees Fahr. (705 degrees Cent.) by H. J. French,
- (b) rate past 1000 degrees Fahr. (540 degrees Cent.) by E. C. Bain,
- (c) half temperature-time, by M. A. Grossmann,

and to these may be logically added,

- (d) rate past the knee of the S-curve (this temperature may depart considerably from 1000 degrees Fahr. (540 degrees Cent.) due to the large quantities of alloying elements in the air hardening steels).

To determine which hardenability parameter leads to the best agreement between the hardness at a given location on the hardenability specimen and the hardness in the size round (A/V) having the same cooling rate past the temperatures of 600, 1000, and 1300 degrees Fahr. (315, 540 and 705 degrees Cent.), comparative tests were made on several air hardening steels (containing about 1.20 per cent carbon, 2 per cent manganese, 1 per cent chromium and 1 per cent molybdenum) as follows:

Hardnesses of these eight experimental air hardening steels were obtained in 4-inch round by 4-inch long cylinders, using a temperature of 1550 degrees Fahr. (845 degrees Cent.) and soaking times of 20 minutes and 2 hours. These data are given in columns 2 and 3, Table II. This size round has an A/V of 1.5. The cooling rates for a 4-inch round by 4-inch long specimen past 1300, 1000 and 600 degrees Fahr. (705, 540 and 315 degrees Cent.) are as follows:

1300 degrees Fahr. (705 degrees Cent.)	58	degrees Fahr. per Minute
1000 degrees Fahr. (540 degrees Cent.)	32	degrees Fahr. per Minute
600 degrees Fahr. (315 degrees Cent.)	12.5	degrees Fahr. per Minute

These rates of cooling will be found on the air hardenability specimen at the following distances from the 6-inch slug:

58	degrees Fahr. per Minute	(1300 degrees Fahr.)	0.12 inch
32	degrees Fahr. per Minute	(1000 degrees Fahr.)	0.60 inch
12.5	degrees Fahr. per Minute	(600 degrees Fahr.)	2.00 inches

In other words, the above distances are those at which the hardenability specimen cools like a 4-inch round by 4-inch long piece of steel past the temperatures of 600, 1000 and 1300 degrees Fahr. (315, 540 and 705 degrees Cent.). The upper nose on the S-curve for these steels is probably close to 1150 degrees Fahr. (620 degrees Cent.).

Table II
A Comparison of Hardness on Hardenability Specimen and Actual Size Rounds, Based Upon Equal Cooling Rates Past 600, 1000 and 1300 Degrees Fahr.

Steel	Treating Temps. Deg. F.	Re in 4" Rd.		Rockwell C at			Difference in Re at		
		20 Min.	2 Hr.	0.12"	0.60"	2.00"	0.12"	0.60"	2.0"
		Soak	Soak	Degrees Fahr.			Degrees Fahr.		
L	1550	60	60	1300	1000	600	1300	1000	600
M	1550	62	63	56.5	60	62.5	-3.5	0	+2.5
N	1550	61	59	61	62	64	-1.0	0	+1.0
O	1550	63	62	57	59.5	62	-2.0	0	+1.0
P	1550	63	62	61	62.5	63	-1.0	0	0
Q	1550	64	63	63.5	62.5	62.5	0	-0.5	-0.5
R	1550	57	58	53	56	60.5	-4.0	-1.0	+2.5
S	1550	63	62	62	61	61.5	0	-1.0	-0.5
S	1550	62	63	59.5	61	63.5	-2.5	-1.0	+0.5
		2.10 Per Cent C,		12.5 Per Cent Cr,	1.0 Per Cent Mo		Steel		
				0.12"	0.65"	2.00"	0.12"	0.65"	2.00"
				Degrees Fahr.			Degrees Fahr.		
C	1750	45 Min. Soak		1300	1000	600	1300	1000	600
		44/45		45.5	46	61.5	+0.5	+1.0	+16.5
				0.40"	0.80"	2.00"	0.40"	0.80"	2.00"
				Degrees Fahr.			Degrees Fahr.		
C	1800	45 Min. Soak		1300	1000	600	1300	1000	600
		50		49	51	62	-1.0	+1.0	+14

Measurements of Rockwell "C" hardness on the hardenability specimens of these eight experimental steels at the locations given above were made and are shown in columns 4, 5 and 6, Table II. Columns 7, 8 and 9 show the difference between the hardness at these three locations and the range of hardness found in the 4-inch round by 4-inch long specimen for the 20-minute and 2-hour soaks. These data indicate closest agreement when the cooling rate past 1000 degrees Fahr. (540 degrees Cent.) is chosen as the hardenability parameter. The 1300-degree Fahr. (705-degree Cent.) parameter results in hardnesses on the hardenability specimen which are too low and the 600-degree Fahr. (315-degree Cent.) parameter results in hardnesses on the specimen which are too high, thereby indicating that best agreement can be had by using a parameter somewhere between 600 and 1300 degrees Fahr. (315 and 705 degrees Cent.). For this reason 1000 degrees Fahr. (540 degrees Cent.) was chosen, and is used here in the following experiments. Fortunately the cooling rate gradient past 1000 degrees Fahr. (540 degrees Cent.) is less steep than that past 1300 degrees Fahr. (705 degrees Cent.) This facilitates locating the hardness position on the hardenability specimen, for as has been shown, the cooling of a 4-inch diameter by 4-inch mass corresponds to a distance of only 0.12 inch from the 6-inch slug when the rate past 1300 degrees Fahr. (705 degrees Cent.) is used, but is 0.60 inch when 1000 degrees Fahr. (540 degrees Cent.) is used. (Treating temperature 1600 degrees Fahr.).

Other air hardening steels, such as the high-carbon, high-chro-

mium steels, have the knee of the S-curve located at higher temperatures than that of the above experimental steels (Mn-Cr-Mo steels) and perhaps another parameter would be more suitable for this type of steel. To determine this, similar comparison tests were carried out on Steel C (Table IV). This is usually thought of as a deep oil hardening steel, and so is more sensitive to the influence of size on the resulting hardness. Such a steel amplifies the error in hardness comparisons between the hardenability test specimen and ac-

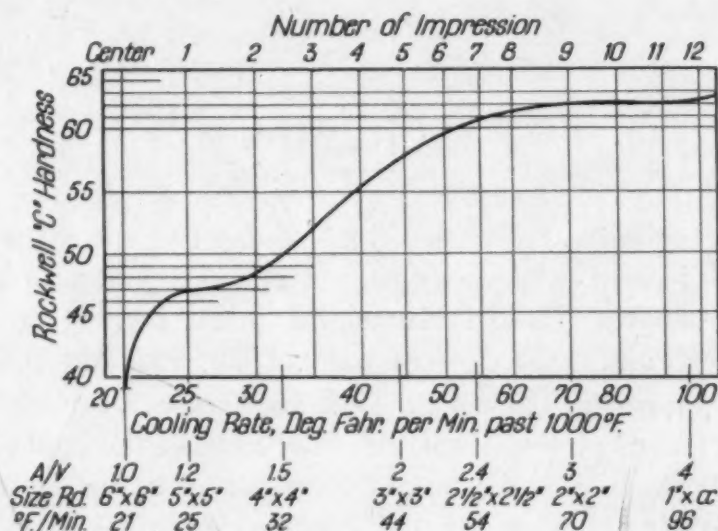


Fig. 6—Air Hardenability Chart.

tual rounds if the wrong hardenability parameter is chosen. These data on the high-carbon, high-chromium Steel C also are shown in Table II. It will be observed that 600 degrees Fahr. (315 degrees Cent.) is too low a temperature upon which to base a parameter for hardenability, while either the 1000 or 1300-degree Fahr. (540 or 705-degree Cent.) parameter would be satisfactory.

A form of air hardenability chart, based upon the cooling rate past 1000 degrees Fahr. (540 degrees Cent.) as the hardenability parameter, is shown in Fig. 6. 14 Rockwell "C" hardness measurements are made along the length of the ground hardenability specimen. These measurements are made at locations corresponding to 5-degree-Fahr.-per-minute cooling rate intervals from 25 degrees Fahr. per minute to 60 degrees Fahr. per minute, and to 10-degree Fahr.-per-minute intervals from 60 degrees Fahr. per minute to 100 degrees Fahr. per minute. Hardnesses are also measured at inside end of specimen and at point of entry into the 6-inch round by

Table III
Locations for Measuring Hardness on Hardenability Specimen Depending on
Initial Heating Temperature

Number of Impression	Cooling Rate °F./Min. Past 1000 °F.	Location in Inches on Hardenability Specimen Where This Cooling Rate Occurs Using Initial Temperatures of—		
		1600 °F.	1750 °F.	1900 °F.
Center	21	Center	Center	Center
1	25	0	0	0
2	30	0.45	0.50	0.62
3	35	0.75	0.85	1.00
4	40	1.00	1.13	1.35
5	45	1.22	1.38	1.64
6	50	1.44	1.60	1.87
7	55	1.65	1.78	2.07
8	60	1.82	1.98	2.27
9	70	2.16	2.31	2.61
10	80	2.47	2.62	3.00
11	90	2.78	3.00	3.40
12	100	3.25	3.48
13	105	3.60	3.85

6-inch long cylinder. The positions along the specimen at which these cooling rates occur vary with treating temperature, as mentioned previously. These locations for initial heating temperatures of 1600, 1750 and 1900 degrees Fahr. (870, 955 and 1040 degrees Cent.) are shown in Table III. The hardnesses developed at these locations are then plotted logarithmically against the cooling rate.

The above described test has been found to be useful in conducting investigations on the hardenability of air hardening steels. The test, however, is too laborious for routine investigation of steels as is done with the oil and water hardening grades. As a matter of fact, this is not necessary in air hardening steels, because it is generally sufficient to obtain the hardenability for a type analysis. Due principally to the high alloy concentration found in air hardening steels, the variation in hardenability from heat to heat is negligible.

PART III

THE EFFECT OF SEVERAL HEAT TREATING VARIABLES ON AIR HARDENABILITY

1. *Effect of Treating Temperature on Air Hardenability*—The effect of treating temperature was determined on the two air hardening steels, A and B, and the high-carbon, high-chromium Steel C. The hardenability assembly was placed in the furnace at 1300 degrees Fahr. (705 degrees Cent.) and allowed to heat up with the furnace in order to minimize differences in heating rates due to the difference in size of the 1-inch round specimen and the 6-inch slug.

The assembly was held at heat for 45 minutes, using the following temperatures:

Steel	Temperatures
A	1700, 1750, 1800, 1850 Degrees Fahr.
B	1800, 1850, 1875, 1900 Degrees Fahr.
C	1700, 1750, 1800, 1850 Degrees Fahr.

The assembly was then removed from the furnace and air-cooled. The results are shown in Fig. 7.

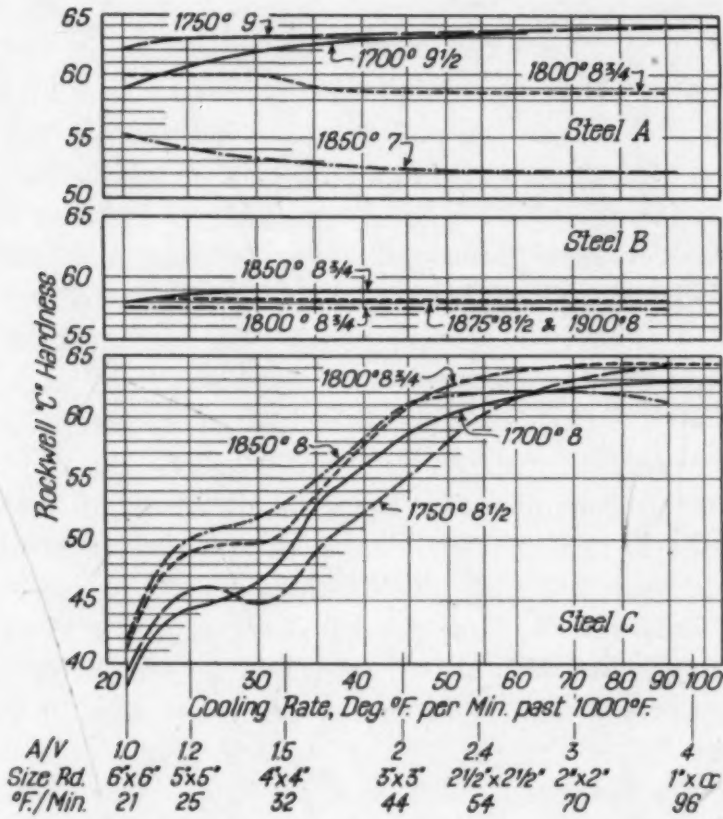


Fig. 7—Effect of Treating Temperature on Air Hardenability of Steels A, B and C. Treating Temperature and Fracture Grain Size Shown on Each Curve.

Steel A (Fig. 7) shows a maximum hardness and greatest hardenability using a treating temperature of 1750 degrees Fahr. (955 degrees Cent.). The center hardness of pieces having an A/V of 1 (6-inch round by 6 inches long) is then 62 R_c. The lower treating temperature of 1700 degrees Fahr. (925 degrees Cent.) appears to be satisfactory for pieces having an A/V of 1.75 or more, but in large sizes, the hardness drops off to 59 R_c at A/V = 1 when treating from this lower temperature. When treating from the higher temperature of 1800 degrees Fahr. (980 degrees Cent.), enough aus-

tenite is apparently retained to drop the maximum hardness to 60 R_c or lower. This maximum hardness of 60 R_c is obtained with the larger sizes ($A/V = 1$), whereas at $A/V = 4$ it drops to 59 R_c indicating that the slower cooling favors the decomposition of the austenite to martensite. Still more austenite is retained by treating from 1850 degrees Fahr. (1010 degrees Cent.), and like treating from 1800 degrees Fahr. (980 degrees Cent.), the maximum hardness of 55 R_c is obtained at low A/V values, and the hardness drops with increasing A/V to 52 R_c at $A/V = 4$.

Steel C, Fig. 7, is considered to be oil hardening, but the hardenability curves show that it will air harden to 63 R_c or better in sizes up to $2\frac{1}{2}$ inches round by $2\frac{1}{2}$ inches long ($A/V = 2.4$ or more) when a treating temperature of 1800 degrees Fahr. (980 degrees Cent.) is employed. Higher hardness, 64 R_c , may be obtained for small sizes (1 inch round or less) by treating from 1750 degrees Fahr. (955 degrees Cent.) instead of 1800 degrees Fahr. (980 degrees Cent.). This is in agreement with the recommended oil treating temperature for this steel; e.g., oil-quenching from 1750 degrees Fahr. (955 degrees Cent.) yields a maximum hardness of 65 to 66 R_c . 1850 degrees Fahr. (1010 degrees Cent.) appears to be too high a treating temperature even for air treatment, because sufficient austenite is retained to decrease the maximum hardness to 62 R_c . The 1850-degree Fahr. (1010-degree Cent.) treating temperature does, however, increase the hardness of larger sizes ($A/V = 2$ or less), because, with the more stable austenite formed, transformation does not take place in the pearlite zone, but more so at temperatures below the "M" point than is permissible with lower treating temperatures.

Steel B (Fig. 7) is shown by the hardenability curves to be extra deep hardening and easy to heat treat. This steel, incidentally, is noted as an exceptionally tough air hardening steel. As shown in Fig. 7 it air hardens to 57 to 59 R_c in all sizes from $A/V = 4$ to $A/V = 1$; i. e., from sizes 1 to 6 inches in diameter. The best treating temperatures appear to be 1875 degrees Fahr. (1025 degrees Cent.) for air-treating larger sizes and 1850 degrees Fahr. (1010 degrees Cent.) for treating smaller sizes (4 inches in diameter or less). Treating from 1800 degrees Fahr. (955 degrees Cent.) results in insufficient carbide solution and slightly lower hardness. Likewise, the 1900-degree Fahr. (1040-degree Cent.) treatment results in hardnesses slightly low, probably due to the retention of austenite.

2. *Effect of Time at Treating Temperature on Air Hardenability*—This series of tests was carried out for two reasons:

- (a) To determine the effect of soaking time on the hardenability of several steels,
- (b) To determine what effect this soaking time had on the hardenability procedure, inasmuch as the assembly varies from 1 to 6-inch round cylinders, and consequently the soaking times are different for various parts.

One would normally expect that increased soaking time would act similar to increased treating temperature. Therefore, if the treating temperature at which the soaking time is being investigated is lower than that which produces maximum hardness, the longer soaks should increase the hardness; conversely, if the steel is treated at the temperature which produces maximum Rockwell hardness, longer soaks would probably decrease the hardness due to the greater solution of alloying elements and the retention of more austenite.

Tests to determine the effects of soaking times were carried out on Steels A, B and C, previously mentioned. These steels were held for $\frac{1}{2}$, 1 and $4\frac{1}{2}$ hours at heat, using temperatures of 1750 degrees Fahr. (955 degrees Cent.) for Steels A and C, and 1875 degrees Fahr. (1025 degrees Cent.) for Steel B. The hardenability curves obtained are shown in Fig. 8.

As shown in Fig. 8C, which relates to the high-carbon, high-chromium Steel C, the $4\frac{1}{2}$ -hour soak resulted in somewhat greater hardness for the larger sizes ($A/V = 2.5$ or less) than the $\frac{1}{2}$ or 1-hour soak. There is not much difference in hardness between the $\frac{1}{2}$ -hour and the 1-hour soaks.

Fig. 8A shows the effect of soaking time on the hardenability of Steel A. Although the effect is small, the shorter soaking time of $\frac{1}{2}$ hour results in an increase of about 1 R_c point in hardness over that obtained by soaking for $4\frac{1}{2}$ hours. Soaking for 1 hour results in intermediate hardnesses. As shown in Fig. 7 increasing the treating temperature of this steel above 1750 degrees Fahr. (955 degrees Cent.) results in the retention of considerable austenite with a decrease in hardness. Thus, the longer soaking time of $4\frac{1}{2}$ hours is in agreement with the statement that the longer hold would be similar to increasing the treating temperature.

Fig. 8B which relates to Steel B shows that soaking time has practically no effect upon the hardenability. This is in agreement with the results found in Fig. 7 to the effect that change in the treat-

ing temperature has practically no influence upon the hardenability.

These tests on the effect of soaking time on air hardenability show that extremely long soaks are equivalent to an increase in treating temperature. However, there is little difference in hardenability when one compares the results obtained from $\frac{1}{2}$ -hour to 1-hour soaks. The significance of these results is that the differences in

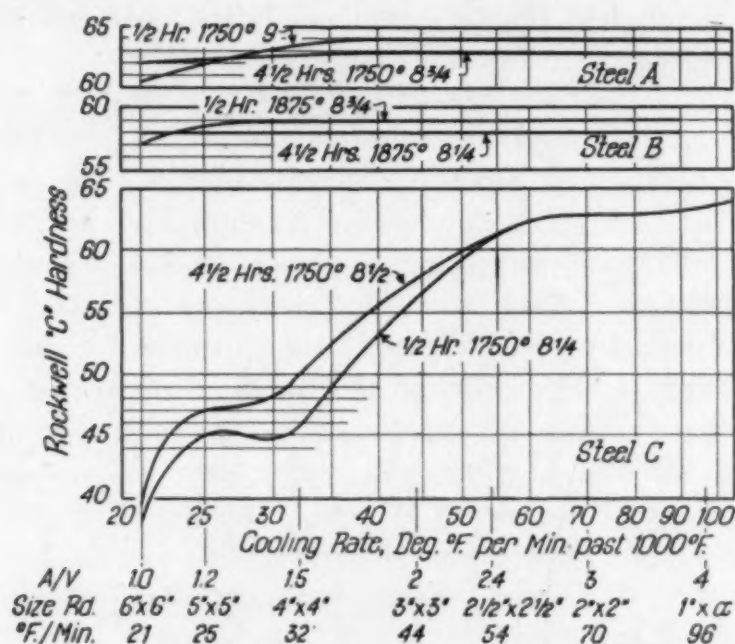


Fig. 8—Effect of Soaking Time on Air Hardenability of Steels A, B and C.

soaking time to be found in the different parts of the hardenability assembly would have little effect on the hardenability results.

3. *Rate of Heating Through the Critical*—Due to the fact that the hardenability assembly consists of a 6-inch diameter section and a 1-inch diameter section, the latter would naturally heat faster through the critical range, and the following tests were carried out on Steels A and B to determine whether the rate of heating through the critical range has an effect on hardenability.

Steel A was heated slowly through the critical as follows: The gas furnace used was set at 1300 degrees Fahr. (705 degrees Cent.), the assembly placed in the furnace, allowed to come to temperature, and then slowly heated with the furnace to 1600 degrees Fahr. (870 degrees Cent.). Following this, the assembly was heated in the normal manner to 1750 degrees Fahr. (955 degrees Cent.), held at heat for 30 minutes, and air-cooled. To obtain fast heating the gas fur-

nace was set at 1600 degrees Fahr. (870 degrees Cent.), the 6-inch slug placed in the furnace and heated to 1600 degrees Fahr. (870 degrees Cent.). After the 6-inch slug was heated to 1600 degrees Fahr. (870 degrees Cent.), the hardenability specimen was placed in the furnace and heated to 1600 degrees Fahr. (870 degrees Cent.),

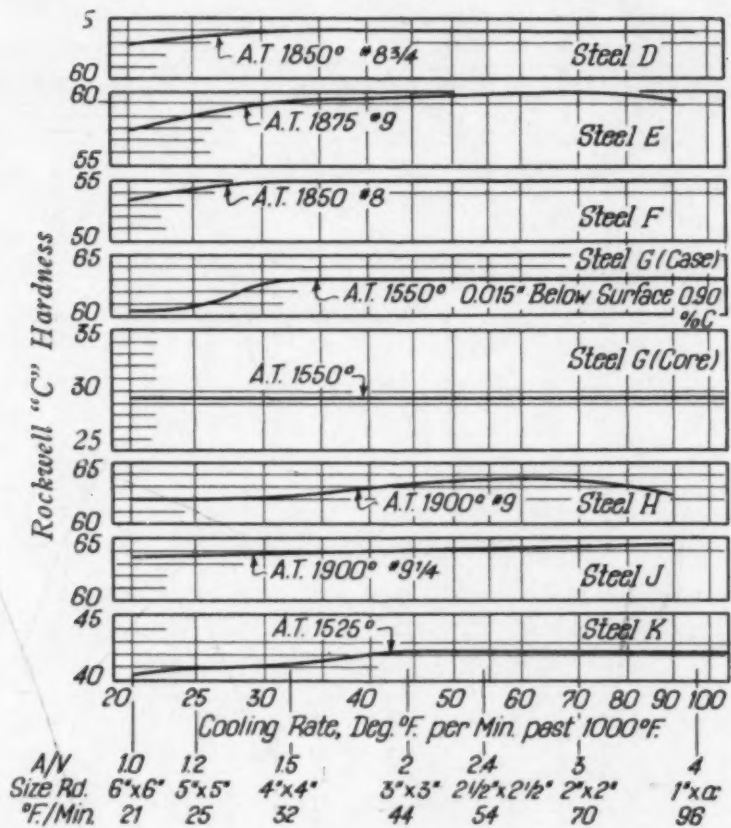


Fig. 9—Hardenability Curves of Several Air Hardening Steels.

and while hot it was screwed into the slug. Following this, the assembly was heated to 1750 degrees Fahr. (955 degrees Cent.), held at heat for 30 minutes, and air-cooled. It was found that the rate of heating had no effect on the hardenability.

Steel B was similarly treated using a temperature of 1350 degrees Fahr. (730 degrees Cent.) instead of 1300 degrees Fahr. (705 degrees Cent.); 1650 degrees Fahr. (900 degrees Cent.) instead of 1600 degrees Fahr. (870 degrees Cent.); and 1850 degrees Fahr. (1010 degrees Cent.) instead of 1750 degrees Fahr. (955 degrees Cent.). It was again found that rate of heating through the critical had no effect on the hardenability.

4. Hardenability of Typical Air Hardening Steels—The air

Table IV
Analyses, Treating Temperatures and Hardnesses Attainable in Various Air Hardening Steels As a Function of A/V

Steel Designation	Per Cent						Hardening Temp. °F.	Minimum A/V for Min. Re of						
	C	Mn	Si	Cr	Ni	W		Mo	64	62	60	55	50	45
A	0.93	0.62	0.24	5.04	0.20	...	0.93	3.0 3.0	1.4 1.0	1.1 <1.0/ <1.0/	<1.0 <1.0
B	0.39	0.40	0.99	5.10	0.09	...	1.22	no	no	no	<1.0
C	2.16	0.40	0.40	12.00	0.48	...	0.087	no	no	no	<1.0	...	1.3	1.1
								3.8	2.7	2.2	1.7	1.6	1.4	1.1
								2.6	2.1	2.0	2.0	1.5	1.1	1.0
									2.3/ 3.3	2.0	1.6	1.2	1.1	1.0
D	1.50	0.35	0.36	12.09	0.35	...	0.83	no	<1.0
E	1.13	0.42	0.34	16.70	0.36	0.02	...	1.6	<1.0
F	0.35	0.41	0.40	12.90	0.50	...	0.26	no	no	1.41	<1.0
G*	0.104	0.36	0.26	1.54	3.67	no	no	<1.0
H	0.57	0.56	0.71	7.45	0.22	7.77	0.15	no	1.3	<1.0
I	0.71	0.47	1.17	3.04	0.33	...	5.63	2	1.0	<1.0
J	0.26	0.49	0.17	1.24	4.28	no	<1.0
K	0.69	2.09	0.35	1.08	0.15	...	1.57	no	no	no	no	no	no	<1.0
T								<1.0

*Carburized 1650° F. 8 hrs.; hardness taken 0.015" below surface at about 0.90% C.

hardenability of some typical air hardening steels (A to K inclusive) is shown in Table IV, together with analyses, and heat treatment employed. The hardenability curves of Steels D, E, F, G, H, J and K are shown in Fig. 9; those of the other steels in Table IV have been presented in Figs. 6 and 7. Table IV shows the minimum A/V (largest regular size) which can be hardened to a given R_c hardness for a given heat treating procedure. To illustrate this table, the hardenable stainless steel, E (Type No. 440), can be air hardened from 1875 degrees Fahr. (1025 degrees Cent.) to 60 R_c in all sizes smaller than that which has an A/V of 1.4. This A/V value corresponds to a cylinder about 4 inches in diameter by 4 inches long, or a 4-inch cube. All sizes smaller than this will harden to at least this Rockwell hardness. This is true because none of the steels listed in Table IV retain more austenite in sizes down to 1-inch round than they do in 6-inch rounds.

In applying the data of Table IV it will suffice to make an estimate of the A/V value for the piece to be heat treated. In most cases it will generally suffice to neglect small holes, protrusions and small recesses in calculating the A/V value for a die or a tool. In the case of large holes in the tool which contribute substantially to the calculated A/V value these should obviously be included in the calculation. In general, the variation in air hardenability of an air hardening steel is negligible from heat to heat for a given type analysis; consequently the data of Table IV should lead to accurate estimates of the hardness to be expected in various size and shape tools made from these types of steels.

SUMMARY

1. Experimental data are presented to show that the center cooling velocity of regular shapes is controlled by the area per unit volume, A/V , relationship, and is independent of treating temperature.
2. A gradient air hardenability test is described. This test enables a determination of the air hardening properties of a given air hardening steel to be made, ranging from the equivalent of a 1-inch round by 8-inch long cylinder to a 6-inch round by 6-inch long cylinder ($A/V = 4.25$ to $A/V = 1$).
3. Air hardenability is stated here in terms of the cooling velocity (degrees Fahr. per minute) with which the steel must be cooled

past 1000 degrees Fahr. (540 degrees Cent.) to obtain a given R_c hardness. The rate past 1000 degrees Fahr. (540 degrees Cent.) leads to a better correlation between the hardenability specimen and actual rounds and shapes, than the rate past 1300 degrees Fahr. (705 degrees Cent.), or that past 600 degrees Fahr. (315 degrees Cent.)

4. Small changes in hardening temperature have an effect on the hardness of the high-carbon (over about 1 per cent) air hardening steels, but have little effect on the hardness of the lower carbon steels. Small differences in soaking time, such as between $\frac{1}{2}$ hour and 1 hour, appeared to have little effect on the hardness obtained. Differences of 1-hour and 4-hour soaking times have an effect similar to increasing the treating temperature.

5. The rate of heating through the critical has a negligible effect on air hardenability probably due to the large quantity of alloying elements present in the air hardening steels.

6. In most cases it is possible to make a good approximation of the A/V value of a tool to be heat treated. Combining this value with the hardenability of the steel to be used, it is a simple matter to estimate with accuracy the hardness to be obtained throughout the tool by air treatment.

7. The hardenability of several typical air hardening steels is presented in Table IV.

In general the variation in air hardenability of air hardening steels is negligible from heat to heat for a given type analysis; consequently the data of Table IV should lead to accurate estimates of the hardness to be expected in various size and shape tools made from those air hardening steels.

ACKNOWLEDGMENT

The generous co-operation of the Staff of the Metallurgical and Chemical Laboratories of The Carpenter Steel Company in carrying out this investigation is gratefully acknowledged. The authors wish to thank Mr. J. H. Parker, president, and Mr. B. H. DeLong, chief metallurgist, of The Carpenter Steel Company, for permission to publish these results.

DISCUSSION

Written Discussion: By Stewart M. DePoy, metallurgist, Delco Products Div., General Motors Corp., Dayton, Ohio.

To the knowledge of the discussor, this is the first work done on the hardenability of air hardening steels. In the past the only method of checking this type of material was a cross section test.

It is unfortunate that the test is as cumbersome as it is. This fact more or less eliminates the test for routine checking of received material. However, as the authors state, this type of material will not vary much in hardenability from heat to heat. This fact, coupled with the information shown in Table IV, makes the results of the paper very useful for the tool maker and tool designer.

It is suggested that the cooling rates be converted from degrees per minute to degrees per second. This would give a ready comparison between air hardening and oil and water hardening steels. For example:

Standard oil hardening tool steels required a cooling rate of approximately 25 degrees Fahr. per second to harden to 60 R_c when austenitized at 1500 degrees Fahr. (815 degrees Cent.). The authors show that Steel C, an oil hardening high-carbon, high-chromium type, requires a cooling rate of about 0.715 degree per second to harden to 60 R_c when austenitized at 1800 degrees Fahr. (980 degrees Cent.). By comparison it may be said that the hardenability of Steel C is about 35 times greater than that of manganese oil hardening steels.

The authors have shown quite conclusively the effect of high austenitizing temperatures on most air hardening steels. It is pointed out that the retained austenite phenomenon is much more pronounced on the 1 per cent carbon, 5 per cent chromium, 1 per cent molybdenum type than on other air hardening steels. This explains the inconsistent results that have been reported from the field on this type of material. However, the authors show that proper austenitizing temperature and time will probably produce as good or better finish structure than can be obtained from the competitive types.

It would be interesting to observe the effect of this hardenability test on high speed steels. Have the authors conducted any such tests, and if so, what were the results?

The authors have contributed a very informative paper to the literature on hardenability. It is also useful to the metallurgist or tool designer for the selection of the proper material for large tools or dies. The A/V method used by the authors is quite clever, and makes it possible for a quick examination of the hardness expectancy of any shape of piece. The authors are to be congratulated on this splendid paper.

Written Discussion: By Arthur E. Focke, research metallurgist, Diamond Chain and Manufacturing Co., Indianapolis.

This very instructive paper attracted our attention because we use air hardening steels in many of our tools. We treat them in an atmosphere controlled furnace and cool them in a water-cooled chamber attached to the furnace in which the cooling rate is supposed to be essentially the same as that obtained in air.

The tools are run on a wire mesh tray so that they are not in direct contact with the water-cooled wall. For some time we have observed that the

hardness we developed from this method, particularly on large sections, was not as high as that which was recorded in the sales literature for the type of steels used, and we have suspected that our cooling rate was not as high as in air.

It was obvious that the data in this paper would make it possible for us to compare very quickly the results from this method with those of a regular air cool. However, in order to be sure that we understand the authors' data and calculations it was necessary to work through them, and for this we found it convenient to remember that for cylinders

$$A/V = \frac{2d + 4L}{dL}$$

in which "d" is the diameter and "L" the length; that if "d" equals "L" then

$$A/V = \frac{6}{L}; \text{ that for parallelopipeds } A/V = \frac{2xy + 2xz + 2yz}{xyz} \text{ where } x, y, \text{ and } z$$

are the length of the sides respectively; and that for a cube $A/V = \frac{6}{x}$.

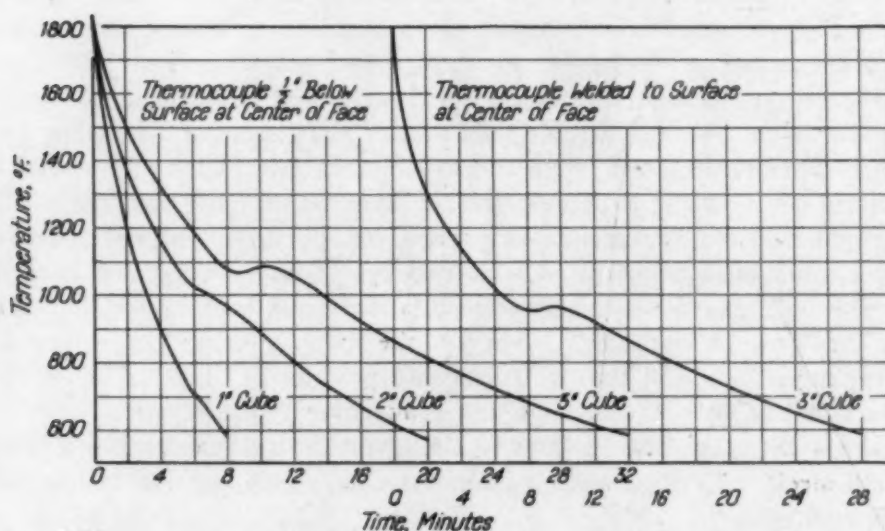


Fig. A—Cooling Curves. Type C Air Hardening Tool Steel.

Unquestionably the authors felt that it was unnecessary to lengthen their paper by the inclusion of such elementary mathematics, but to the writer it seemed desirable to note these relationships for the benefit of other practical heat treaters who may wish to study and use the data of this valuable paper.

To test our equipment we made up a 3-inch, a 2-inch, and a 1-inch cube of the steel identified as type "C" in this paper. These were heated to 1800 degrees Fahr. (980 degrees Cent.) and cooled in the chamber. After hardening, the Rockwell "C" hardness at the center of the face of these cubes was 40-43, 60-62, and 62-64, respectively, before grinding and 38-40, 56-60, and 60-63 after grinding off 0.005 inch. From the 1800-degree Fahr. (980-degree Cent.) curve in Fig. 7, it can be seen that these hardnesses would be expected at cooling rates of slightly less than 20 and about 37 degrees Fahr. per minute

for the 3 and 2-inch cubes; and since the A/V relations for these are 2 and 3 respectively, it would appear that the cooling rate in our equipment is only about one-half of that obtained in air.

This explains why we got only a hardness of about 60 on some rolls which were about 8 inches in diameter and 3 inches thick ($A/V = 1.16$) made from a steel designated as type D in this paper, since the cooling rate in our equipment for a piece this large could be expected to be only about 11 degrees Fahr. per minute (about one-half of 22 shown in Fig. 3 for this A/V ratio) and from Fig. 9 it is evident that full hardness would not be developed at this rate.

However, in this same series we drilled a hole $\frac{1}{2}$ inch deep in the center of one face and in it we inserted a No. 8 gage chromel-alumel thermocouple. The cooling curves for the three cubes are shown at the left in Fig. A. Note that both the 2 and 3-inch cubes show evidence of high temperature transforma-

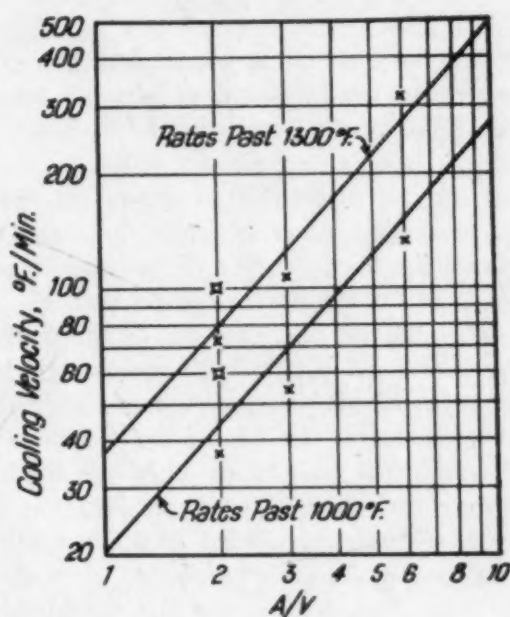


Fig. B—Comparison of Rates in Cooling Chamber with Those in Air.

tion but by extrapolation of these curves and by averaging the actual temperature drop in various time intervals for those which do not show recalescence the following cooling rates were obtained:

Sample	A/V	Cooling Rate (Degrees Fahr. Per Minute)	
		At 1300	At 1000
3" cube	2	73	35
2" cube	3	113	56
1" cube	6	320	140

When these results are compared with the authors' curve of Fig. 3 (recorded as crosses in Fig. B), it would appear that the actual cooling rate in our cooling chamber was only slightly less than in air. To check these results further, each branch of the thermocouple was welded to the surface of the 3-inch cube about $\frac{1}{2}$ inch apart at the center of the face opposite that drilled for the previous test and the sample retreated.

The cooling curve for this test is shown at the right in Fig. A, the apparent cooling rate at 1300 degrees Fahr. (705 degrees Cent.) was about 100 degrees Fahr. per minute and at 1000 degrees Fahr. (540 degrees Cent.) it was about 60 degrees Fahr. per minute and these results are shown as circled crosses in Fig. B. The surface hardness at the center of the faces of this re-treated block was the same as before (38 to 44 Rockwell "C").

It is possible that our method of measuring the temperature was not correct but if one accepts these cooling rate data it is necessary to look for some other explanation for the low hardness values.

While these contradictory results are confusing, we feel that this paper has given us a tool with which to analyze our results critically and we sincerely appreciate the time and effort which the authors expended to make it available to us.

Authors' Reply

Mr. DePoy points out that expressing the cooling rates in degrees per second would be more convenient for comparative purposes than expressing them in degrees per minute. The authors agree that there is considerable merit to this suggestion but have used minute units in order to avoid expressing the rates in decimals. The method and example shown for comparing the hardenability of oil and air hardening steels is interesting, and the statement that Steel C has about 35 times the hardenability of manganese oil hardening steels demonstrates that Steel C is on the borderline between air hardening and oil hardening steels. Up until the present time no air hardenability tests of the nature described have been carried out on high speed steels but substitute tests are contemplated. The extremely high temperatures employed in treating high speed steel would be detrimental to the life of the 6-inch round employed but this difficulty could be minimized by heating only the hardenability specimen to the treating temperature and then screwing it into the 6-inch round slug portion at some lower temperature, say around 1600 degrees Fahr. (870 degrees Cent.), and from there allowing the entire assembly to cool to room temperature. Perhaps a better method of determining the hardenability of these high temperature steels is to cool them in a controlled-rate cooling furnace at various rates past 1300 and 1000 degrees Fahr. (705 and 540 degrees Cent.) as it has been shown that it is the rates of cooling past these temperatures which determine the hardness of the steels. This cooling rate would be employed only within the close vicinity of 1000 to 1300 degrees Fahr. (540 to 705 degrees Cent.). After passing through this narrow range the small specimen used could be air-cooled. The authors appreciate Mr. DePoy's interest in this work and wish to thank him for his constructive criticism.

The authors were greatly interested in the description of the simulated air-cooling equipment used by Dr. Focke and were pleased to learn that this paper was of some value in his work. There appears to be no explanation as to why the hardnesses obtained by Dr. Focke on Steel C differ from those presented by the authors. It may be well to point out that Steel C is what would be considered a shallow air hardening or deep oil hardening steel, and it may be the difference in analysis and grain size of this type of steel together with the marked change in hardenability with temperature, especially between the

treating temperatures of 1750 and 1800 degrees Fahr. (955 and 980 degrees Cent.), as shown in Fig. 7, would cause appreciable differences in the hardnesses obtained in various size rounds. Up to the present time the authors have run only a limited number of hardenability tests on this steel and unfortunately there is not sufficient time available before the publication deadline to carry out more tests to ascertain the variation in hardenability with changes in analysis. However, more tests on this steel are contemplated. Dr. Focke's data show that the cooling rate of the 3-inch square is 100 degrees Fahr. per minute past 1300 degrees Fahr. (705 degrees Cent.) and 60 degrees Fahr. per minute past 1000 degrees Fahr. (540 degrees Cent.), and that these values are appreciably faster than obtained on the inside of the same slug; namely, 73 and 35 degrees Fahr. per minute respectively. This is in variance with the results obtained by the authors but a difference in technique perhaps accounts for the difference in results. The authors placed the thermocouple bead under the surface of the metal, whereas Dr. Focke welded the thermocouple wires directly on to the surface. The authors were reluctant to use the latter method because of a possible tendency of the junction to cool faster than the surface of the metal. The reduced formula which Dr. Focke presents for the determination of A/V will no doubt be a help to those who wish to apply the material presented in the paper or carry out further experiments. Dr. Focke's discussion indicates that he has spent considerable time on the subject of air hardenability and the authors appreciate his discussion.

THE PARTITION OF MOLYBDENUM IN STEEL AND ITS RELATION TO HARDENABILITY

BY FRED E. BOWMAN

Abstract

The investigation of the partition of molybdenum between ferrite and carbide formed isothermally from austenite and by tempering martensite has been extended to 1200 and 1100 degrees Fahr. (650 and 595 degrees Cent.). Molybdenum segregates to the carbide when the isothermal transformation takes place at these temperatures. The complex carbide $(Fe, Mo)_{23}C_6$, previously reported at 1300 degrees Fahr., continues to form at lower subcritical transformation temperatures.

An explanation for the increased hardenability produced in steels by molybdenum is offered on the basis of the effect of this segregation and altered carbide structure on nucleation rates.

The alpha iron lattice parameter versus molybdenum content relationship is extended to the room temperature solid solubility limit (6 per cent molybdenum).

MUCH progress has been made in developing a mechanical description of the action of the carbon atom in the process of hardening steel, but not nearly as much clarity has been achieved in the description of the part third elements play in co-operation with the carbon atom to increase the hardenability of steel. This report is the seventh of a series (1) to (6)* intended to determine the kinetics of the molybdenum atom during the hardening process.

Early attempts to explain the effect of third elements on hardenability postulated that the added element retarded the rate of diffusion of carbon in austenite. Wells and Mehl (7) have shown for nickel and manganese, and Ham, Parke, and Herzig (3) have shown for molybdenum, that the effect of these elements upon the rate of diffusion of carbon in austenite cannot account entirely for their influence upon the rate of transformation of austenite.

*The figures appearing in parentheses pertain to the references appended to this paper.

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The diffusion of the alloying element remained as the most obvious explanation for the increased hardenability produced by the alloying element. The decomposition of austenite, at temperatures above that at which martensite is formed, involves the formation of two constituents, ferrite and carbide. If the diffusion of the alloying element in austenite is the factor determining the rate at which the austenite transforms, the alloy content of one or the other of these constituents must be higher than that of the parent phase. In this report it will be shown that molybdenum does diffuse to the carbide during transformation of austenite even at subcritical temperatures as low as 1100 degrees Fahr. (595 degrees Cent.). It was shown previously (5) that the carbides formed by isothermal transformation at 1300 degrees Fahr. contained a major portion of the total molybdenum originally present in the austenite.

In that temperature region where molybdenum displays its greatest influence, austenite decomposes into a lamellar aggregate of ferrite and carbide. This transformation proceeds through a process of nucleation and growth and the nucleating phase, as determined by metallographic observation, is the carbide. The fact that these carbides contain a large portion of the total molybdenum present indicates that the partition of this element is related to its effect upon hardenability.

EXPERIMENTAL PROCEDURE AND RESULTS

The results of a study of the partition of molybdenum which occurs during the isothermal transformation at 1300 degrees Fahr. (705 degrees Cent.) of pure iron-molybdenum-carbon alloys of approximately eutectoid carbon content have been reported earlier (5). That report also included the data obtained from long-time tempering treatments of the martensitized alloys. The present work is concerned with an extension of the study to transformation temperatures down to 1000 degrees Fahr. (540 degrees Cent.).

A new series of alloys was prepared, closely approximating the composition of the eutectoid alloys used in the earlier work. As previously, these alloys were prepared by vacuum melting electrolytic iron, pure molybdenum powder, and carbon. Developments in technique, however, permitted the new alloys to be vacuum cast into a 1-inch internal diameter thick-walled steel mold. As a result, less segregation of molybdenum was encountered in these ingots than in

the earlier ones, which had been allowed to solidify in the crucible. After being forged into strips $\frac{1}{8}$ by 1 inch in cross section the materials were homogenized in vacuum for 50 hours at 2200 degrees Fahr. (1205 degrees Cent.). Newly determined diffusion data (6) indicated the advantage of the higher homogenization temperature over the treatment at 2000 degrees Fahr. (1095 degrees Cent.) used formerly. No decarburization could be detected metallographically.

Times for the completion of the subcritical transformation of austenite in the various alloys were determined metallographically from samples austenitized in a deoxidized lead bath at 1600 degrees Fahr. (870 degrees Cent.) and then quenched into lead baths and held for various lengths of time at 1200, 1100, and 1000 degrees Fahr. (650, 595, 540 degrees Cent.).

Attempts to separate the carbides electrolytically from material transformed at 1200 degrees Fahr. (650 degrees Cent.) were unsuccessful. Although the results obtained by identical methods from alloys transformed at 1300 degrees Fahr. (705 degrees Cent.) were excellent, the increased fineness of the carbides formed at the lower temperature greatly increased their reactivity toward the electrolyte.

The X-ray apparatus and techniques employed in the investigation of carbide structures and in determining the partition of molybdenum have been described previously (5). Since the first report on the effect of dissolved molybdenum on the lattice parameter of alpha iron (4) an opportunity has arisen to extend these measurements to the approximate limit of room temperature solid solubility, or to 6 per cent molybdenum. In the course of this work, corrections were made in the a_0 values of the two highest molybdenum alloys previously reported.¹ No occasion had arisen to use the values from these two alloys in determining the partition of molybdenum. The revised and extended lattice parameter versus molybdenum content relationship is shown in Fig. 1.

Samples austenitized at 1600 degrees Fahr. (870 degrees Cent.) and completely transformed at 1200 degrees Fahr. (650 degrees Cent.) in a lead bath were prepared for X-ray examination. Scale

¹It was found that the stress-relieving temperature selected for the alloy samples had an appreciable effect upon the lattice parameter. Stress lowered the a_0 value, and this effect was most pronounced in the higher molybdenum alloys. One hour at 2000 degrees Fahr. (1095 degrees Cent.) was necessary to anneal the filings completely in the case of the 6 per cent molybdenum alloys.

This sluggishness of iron-molybdenum alloys in recovering from lattice distortion is significant. Wilson and Thomassen (8) have reported that there is a correlation between creep strength and the times and temperatures necessary to produce sharp diffraction patterns in cold-worked alloys. The improvement in creep resistance imparted to steels by molybdenum is probably related to the resistance to stress relief noted in the binary alloys.

was removed and the surface was deeply etched in dilute nitric acid. Diffraction patterns were made in a Debye-Scherrer type camera with the sample arranged to intercept the beam with an edge. Ex-

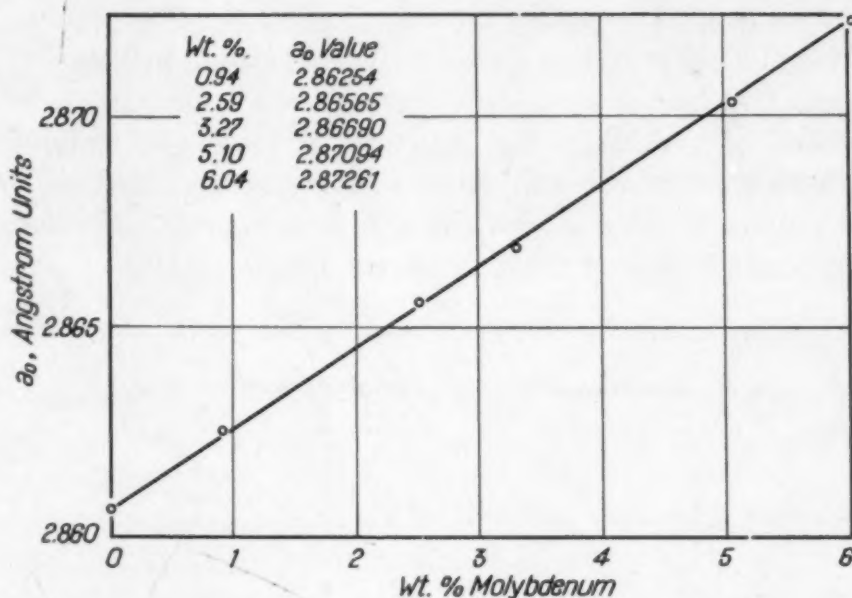


Fig. 1—The Alpha Iron Lattice Parameter as it is Related to Molybdenum Content.

cellent patterns of the carbides were obtained in each case and the structures were found to be identical with those formed at 1300 degrees Fahr. (705 degrees Cent.).² The pattern of the 0.30 per cent molybdenum alloy revealed the orthorhombic structure of cementite, while those from the alloys containing 0.51, 0.74, and 1.02 per cent molybdenum revealed the presence of the face-centered cubic (Fe, Mo)₂₃C₆.

In the case of the alloys transformed at 1300 degrees Fahr. (705 degrees Cent.) the X-ray method gave data agreeing excellently with those obtained by chemical separation and analysis of the carbide. However, as noted earlier, satisfactory chemical separation of the carbides formed at 1200 degrees Fahr. (650 degrees Cent.) was impossible. Furthermore, the ferrite back-reflection patterns from the alloys transformed at this lower temperature were much more diffuse than those obtained from the material transformed at 1300 degrees Fahr. (705 degrees Cent.). This diffuseness of interference line, which can be due to fineness of the grains, inhomogeneity, or stresses,

²Etching the sample with dilute nitric acid permitted the carbide patterns to be obtained directly from the solid sample. No patterns of the carbide could be obtained from unetched samples.

increased appreciably with the molybdenum content of the alloy. Therefore, the results given in Table I, although the best obtainable, are not of the same degree of accuracy as those reported in the earlier paper (5) for 1300 degrees Fahr. (705 degrees Cent.), but they do permit an estimation of the alloy content of the two phases.

It was found that the carbides formed from martensite tempered at 1300 degrees Fahr. (705 degrees Cent.) were of the orthorhombic variety. Although they increased gradually in molybdenum content with time at the tempering temperature, within 250 hours they did not reach molybdenum contents approaching the values obtained in a much shorter time by direct transformation. In order

Table I
Alloys Isothermally Transformed at 1200 Degrees Fahr.

Alloy No.	Per Cent		Transformation Time, Seconds	Per Cent Mo in Ferrite	Type of Carbide	Per Cent Mo in Carbide (Calculated)
	Mo	C				
54	0.30	0.78	250	0.19	Orthorhombic	1.11
71	0.51	0.74	600	0.10	Face-centered cubic	3.04
72	0.74	0.74	1500	0.26	Face-centered cubic	3.73
38	1.02	0.80	2500	0.37	Face-centered cubic	4.64

to determine the extent of molybdenum concentration which would take place in similar periods of time at 1200 degrees Fahr. (650 degrees Cent.), small samples of each homogenized alloy were quenched from a lead bath at 1600 degrees Fahr. (870 degrees Cent.) into water. After removal of scale the samples were placed in a vacuum furnace and held for 150 hours at 1200 degrees Fahr. (650 degrees Cent.). It was found, by metallographic examination after this treatment, that all samples had completely graphitized. No graphitization had been encountered at 1300 degrees Fahr. (705 degrees Cent.) even after 250 hours. New samples were similarly tempered for 100 hours at 1200 degrees Fahr. (650 degrees Cent.) and partial graphitization was found in the three lowest molybdenum alloys, the amount of graphitization decreasing with increasing molybdenum. The 1.02 per cent molybdenum alloy was graphite-free, and metallographic examination revealed spheroidized carbides although they were much finer than those found after the 150-hour treatment at 1300 degrees Fahr. (705 degrees Cent.). The undecomposed carbides in all tempered samples yielded the diffraction pattern of the orthorhombic structure. The distribution of molybdenum in the one alloy which did not graphitize is shown in Table II.

Alloys austenitized at 1600 degrees Fahr. (870 degrees Cent.) and completely transformed isothermally at 1100 degrees Fahr. (595 degrees Cent.) were also investigated. The transformation times for the two alloys of highest molybdenum content were not only much greater than at 1200 and 1300 degrees Fahr. (650 and 705 degrees Cent.), but also the microstructures were different. At the higher

Table II
Alloy Quenched and Drawn at 1200 Degrees Fahr. for 100 Hours

Alloy No.	Per Cent		Type of Carbide	Per Cent	
	C	Mo		Mo in Ferrite	Mo in Carbide
38	0.80	1.02	Orthorhombic	0.87	2.09

transformation temperatures the expected lamellar pearlite was formed with increasing fineness as the temperature of formation was lowered. However, at 1100 degrees Fahr. (595 degrees Cent.) an acicular structure was formed in which the carbides were much more massive than those in the coarsest pearlite.

Because of the size of these carbides it was possible to make an electrolytic separation and obtain a chemical analysis, although even here there was considerable decomposition of the carbides in the separation process and no material balance could be obtained. Diffraction patterns of the separated carbides indicated a high predominance of the face-centered cubic material in both cases, but the predominance was much greater in the higher molybdenum alloy. The carbon contents of the carbides supported this evidence. However, the carbide patterns obtained from a sample of the alloy itself indicated a much greater amount of the orthorhombic material than existed in the residue. Apparently the orthorhombic carbide formed at 1100 degrees Fahr. (595 degrees Cent.) along with the face-centered material possesses greater reactivity toward the electrolyte than does the more complex carbide or the orthorhombic material formed at higher temperatures. In the lower molybdenum alloys the carbide structures as obtained from isothermally transformed samples were entirely orthorhombic. These carbides were present as fine pearlite, and no successful separation could be made. The results of both the chemical and X-ray analysis of the alloys isothermally transformed at 1100 degrees Fahr. are listed in Table III.

It will be noted in Table III that the times for complete transformation of the 0.74 and 1.02 per cent molybdenum alloys are much

Table III
Alloys Isothermally Transformed at 1100 Degrees Fahr.

Alloy No.	Per Cent		Transformation Time, Seconds	Carbide Structures		Per Cent Ferrite X-ray	Molybdenum in	
	Mo	C		From Alloy (Percentages Estimated)	From Residue		Carbide Calc.	Carbide Chem.
54	0.30	0.78	90	Ortho.		0.23	0.86
71	0.51	0.74	800	Ortho.		0.31	2.08
72	0.74	0.74	28,800	40% Ortho. 60% F.C.C.	25% Ortho. 75% F.C.C.	0.39	3.12	3.51
38	1.02	0.80	38,000	25% Ortho. 75% F.C.C.	5% Ortho. 95% F.C.C.	0.40	4.40	5.65
Ortho. = orthorhombic, F.C.C. = Face-centered cubic.								

longer than at 1200 degrees Fahr. (650 degrees Cent.) or 1300 degrees Fahr. (705 degrees Cent.). This fact plus the radically different microstructures produced by transformation at 1100 degrees Fahr. (595 degrees Cent.) suggest that the relative importance of the factors determining the transformation process is altered in this temperature range. This suggestion will be discussed further.

Attempts to obtain partition data on alloys quenched and tempered at 1100 degrees Fahr. (595 degrees Cent.) were unsuccessful. Graphitization was found to have occurred in all alloys after 100 hours. Here again the extent of graphitization increased with decreasing molybdenum. Tempering for less time resulted in a ferrite diffraction pattern that was much too diffuse to be of any benefit in obtaining an accurate lattice parameter value. It was found that after 24 and 50 hours at 1100 degrees Fahr. (595 degrees Cent.) the carbides formed from martensite were entirely orthorhombic in all cases.

Isothermal transformation at 1000 degrees Fahr. (540 degrees Cent.) did not yield structures on which measurements of the ferrite lattice constant could be made. In this case even the carbide patterns were not sufficiently complete to permit positive identification. It appears, however, that the orthorhombic material is formed in alloys isothermally transformed at 1000 degrees Fahr. (540 degrees Cent.).

Data have been presented showing the partition of molybdenum resulting from the isothermal transformation of eutectoid austenite containing this element at 1300, 1200, and 1100 degrees Fahr. (705, 650, and 595 degrees Cent.). No definite rule can yet be seen which provides a basis for the quantitative prediction of this partition. A few conclusions can be drawn, however. After isothermal transformation of austenite at the temperatures investigated, molybdenum is present in the carbide in large amounts. The magnitude of the seg-

regation decreases with decreasing transformation temperature. A face-centered cubic carbide which contains at least 3 per cent molybdenum is found. This carbide is formed by the direct transformation of austenite at 1200 and 1300 degrees Fahr. (650 and 705 degrees Cent.) when the austenite contains more than 0.50 per cent molybdenum.

It is hoped that the results of similar investigations of hypo- and hypereutectoid alloys will yield results which will aid in establishing the laws which must govern this partition phenomenon.

DISCUSSION OF RESULTS IN RELATION TO HARDENABILITY

During the transformation of molybdenum-bearing austenite to pearlite the composition and, in some cases, the structure of the carbides formed is greatly altered by the presence of the alloying element in the austenite. It is apparent that the presence of molybdenum in these carbides is essential to their formation. There is now evidence to show that during the transformation of the alloyed austenite to pearlite diffusion of the alloying element takes place and the ultimate rate at which this transformation proceeds is dependent, directly or indirectly, upon the rate of diffusion of molybdenum.

Austenite transforms by a process of nucleation and growth at all subcritical temperatures above that at which martensite is formed. Mehl (9) has shown that both the rate of nucleation and the rate of growth must be combined in the final rate of the transformation itself. Ham (3) has proposed an explanation for the manner in which molybdenum diffusion ultimately determines the rate of growth of the carbide nucleus. Consideration will be given here to the manner in which the partition of molybdenum can affect the rate of nucleation and consequently the extent to which this rate determines hardenability.

According to the nucleation theories outlined by Mehl and Jetter (10), a homogeneous solid solution such as austenite must be considered as being in a constant state of flux with regard to the composition of submicroscopic volumes. It is only when this continuous atomic migration establishes, by chance, a region of the proper composition and size that the nucleus of a new phase can be formed. The continued existence of this nucleus and its ability to grow is dependent upon its having a smaller free energy than the parent phase. This free energy is, in part, a function of nuclear size and it in-

creases with decreasing size, as is pointed out by Sirota (11). Becker (12) has proposed that the number of nuclei formed per second can be expressed in the following manner:

$$N = K e^{-Q/RT} \cdot e^{-A(T)/RT}$$

The $e^{-Q/RT}$ term is recognized as the activation energy factor in the equation for diffusion in solids. The remaining exponential term represents the work necessary to establish the interface between the new and the old phase. This work must be supplied by the free energy difference between the nucleus and the parent phase. For the formation of a stable nucleus, then, a minimum difference in the free energies of the two phases is determined by the work necessary to establish this interface. Since the free energy is dependent upon the size of the nucleus there is a critical size below which, at any given temperature, no nucleus is able to exist permanently.

It can be shown that the free energy of the unstable phase increases more rapidly with decreasing temperature than does that of the precipitating phase. Thus the ΔF change in the transformation of austenite will be greater at, say, 1100 degrees Fahr. (595 degrees Cent.) than at 1300 degrees Fahr. (705 degrees Cent.). The minimum size of a stable nucleus will consequently be smaller and the rate of nucleation will be increased as the temperature decreases.

As the transformation temperature is lowered farther and farther below the critical, the fluctuations in composition of the austenite must simultaneously become less, because of decreased atomic mobility. Consequently, even though the free energy relationships between the austenite and carbide phases are such as to allow a smaller nucleus to be stable and to grow, the probability of its formation will decrease. There must, therefore, be some temperature at which the size of the nucleus that can form (from a standpoint of strict probability) is smaller than that given stability by the difference in free energies. If such a condition exists, the transformation must halt or be carried out by a different process. Some such condition is suggested by the maximum rate of austenite transformation to a soft product which, as is indicated by the Bain S-curves, occurs at from 1100 to 900 degrees Fahr. (545 to 480 degrees Cent.) in plain carbon and low alloy steels. It is in this region, known as the Ar' , that the nucleating phase apparently changes from carbide to ferrite as the transformation temperature is lowered.

The probability of the formation of a stable nucleus decreases

with temperature because of decreased atomic mobility. It is at the same time increased, owing to the smaller nuclear size made stable by the relative changes in free energies of the stable and unstable phases. Thus conditions are established for a maximum in the ultimate rate. From an inspection of the S-curves of plain carbon and low molybdenum steels, it can be seen that this maximum nucleation rate of carbide closely coincides with that of ferrite. However, in higher molybdenum steels another maximum in the rate of austenite transformation is found above the Ar' and is separated from it by a region of decreased rate. Apparently molybdenum raises the temperature of the maximum rate of nucleation by carbide until it no longer coincides with that by ferrite and a temperature range exists in which austenite transformation is nucleated at a rate much slower than the maximum of either nucleating phase.

In the pure iron-carbon-molybdenum alloys studied, such a region of decreased reaction rate was found at 1100 degrees Fahr. (595 degrees Cent.). The microstructures resulting from transformation at this temperature revealed massive carbides, which suggests that the growth rate may have become the controlling rate. The fineness of the carbides formed during transformation above and below this region indicates that the rate of austenite decomposition may be governed at these temperatures by the rate of nucleation.

Once formed, a stable nucleus can grow because of the free energy decrease which results from its increasing size. In the case of eutectoidal decomposition the unstable phase maintains a constant concentration throughout the transformation process and consequently its free energy remains constant. It has been shown by Hull, Colton and Mehl (13) that, in plain carbon steels, while the rate of growth increases slightly with a decrease in transformation temperature, the increase in nucleation rate is tremendous. For instance, in a "pure" iron-carbon alloy containing 0.92 per cent carbon the nucleation rate at 1015 degrees Fahr. (545 degrees Cent.) is some 10^8 times as great as at 1320 degrees Fahr. (715 degrees Cent.). It would appear then that in spite of the decrease in the diffusion rate of carbon with temperature, the increase in transformation rate can be accounted for on the basis of increased nucleation, which in turn, as has been pointed out, is a function of the difference in free energies of the carbide nucleus and the parent austenite.

When, as has been suggested, the maximum nuclear size determined by the probability of its formation becomes less than the stable

size determined by energy relationships, the decomposition of austenite is initiated by ferrite nuclei. Here again similar conditions of free energies must determine the nucleation process. In this case, however, the "fluctuations" in concentration must be such as to allow a sufficiently large area to form free of carbon to provide a nucleus of the stable size. Growth of this nucleus is dependent then upon the rate at which carbon diffuses away from the advancing austenite-ferrite interface. That the conditions governing a eutectoidal reaction in this range of transformation temperatures are no longer in effect is evidenced by the almost complete absence of proeutectoid constituents in hypo- and hypereutectoid alloys allowed to transform to bainite.

The effectiveness of molybdenum in retarding the pearlite transformation has already been pointed out. It remains to fit it into the theories just outlined. The retarding effect of molybdenum on the austenite transformation reaction is brought about by the necessity for segregation of the element in the carbides during transformation.

It must be assumed that the presence of molybdenum in austenite in amounts greater than 0.50 per cent so alters the energy relationships of the phases involved that at certain temperatures the orthorhombic carbide of the Fe_3C type cannot form directly from austenite. The face-centered cubic $(\text{Fe}, \text{Mo})_{23}\text{C}_6$ found in isothermally transformed iron-carbon-molybdenum alloys, containing 0.50 per cent molybdenum or more, has a unit cell volume of approximately 1200 cubic angstroms as compared with about 225 for cementite. The unit cell of the cubic carbide contains 116 atoms in contrast to the 16 in the cementite cell. In addition, Westgren (14) has reported that the molybdenum atoms appear to occupy specific lattice points in the face-centered cubic carbide. It can be seen that the probability of the formation of an $(\text{Fe}, \text{Mo})_{23}\text{C}_6$ nucleus is less than that of the formation of Fe_3C . The present work has been conducted on pure iron-carbon-molybdenum alloys, and direct comparison with alloys of commercial steel analysis may be unwarranted; yet an indication that the presence of molybdenum greatly decreases the rate of nucleation is to be found in some unpublished work carried out in the laboratory of Climax Molybdenum Company. During the isothermal transformation of an austenitized 0.80 per cent carbon, 0.75 per cent molybdenum alloy at 1250 degrees Fahr. (675 degrees Cent.) the rate of nucleation was 267 nuclei per square centimeter per second, while the rate of nucleation of a plain carbon steel at the same temperature

was 24,900 nuclei per square centimeter per second. The delay in the rate of transformation caused by molybdenum cannot be accounted for entirely on the basis of rates of nucleation, however. It has been shown by the S-curves of molybdenum steels (2) that molybdenum also decreases appreciably the rate of growth of the nuclei as compared with the rate in plain carbon steels.

It is not intended that this proposed explanation of the effect of molybdenum upon the rate of transformation of austenite apply to all the elements known to alter the rate of transformation. A common explanation for the effect of an element such as silicon and for molybdenum would, in fact, be surprising. There are several ways in which an alloying element can act to alter the rate of transformation of austenite.

SUMMARY AND CONCLUSIONS

During the transformation of austenite to pearlite, the molybdenum in iron-carbon-molybdenum alloys of approximately eutectoid carbon content segregates in the carbide phase. The magnitude of this segregation decreases with decreasing temperature. The partition, however, never reaches equality, even at temperatures as low as 1100 degrees Fahr. (595 degrees Cent.).

The face-centered cubic $(\text{Fe, Mo})_{23}\text{C}_6$ reported earlier in connection with the transformation of austenite at 1300 degrees Fahr. (705 degrees Cent.) has been found in the same alloy composition range, namely, from 0.50 per cent to 1 per cent molybdenum, after transformation at 1200 degrees Fahr. (650 degrees Cent.). It disappears entirely in the 0.50 per cent molybdenum alloy transformed at 1100 degrees Fahr. (595 degrees Cent.) but is found in combination with the orthorhombic cementite in the higher molybdenum alloys transformed at this temperature. The cementite type carbide probably forms exclusively in all alloys investigated at 1000 degrees Fahr. (540 degrees Cent.).

The segregation of molybdenum during transformation of austenite indicates the necessity for diffusion of this alloying element during the transformation process. Since the diffusion of molybdenum through austenite is much slower than that of carbon, it is obvious that it is this slower rate which will govern the growth rate of the carbide nucleus. In addition, the energy relationships which require the presence of molybdenum in the carbide nucleus and in

some cases which actually complicate the structure of this nucleus, must obviously reduce the probability of its formation.

Thus, an explanation is afforded for the decrease in both the rate of nucleation and rate of growth of carbide nuclei. These rates necessarily determine the hardenability of steel, and the effectiveness of molybdenum in promoting this property is explained.

ACKNOWLEDGMENT

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DISCUSSION

Written Discussion: By G. V. Smith, Research Laboratory, United States Steel Corp., Kearny, N. J.

The writer was particularly interested in the finding that though isothermal transformation at 1300 degrees Fahr. (705 degrees Cent.) resulted in the face-centered cubic carbide $(\text{Fe, Mo})_{23}\text{C}_{14}$, except for the 0.30 per cent molybdenum alloy, samples quenched and tempered at 1300 degrees Fahr. (705 degrees Cent.) showed carbides of the orthorhombic or Fe_3C variety, while at 1200 degrees Fahr. (650 degrees Cent.) the same results were obtained on isothermal transformation, but on quenching and tempering graphitization resulted. Does the author have any thoughts on the question of whether the occurrence of graphitization as described is related to the composition and structure of the carbide or to purely a nucleation effect? In this connection it might be interesting to determine whether a sample which had been quenched and tempered at 1300 degrees Fahr. (705 degrees Cent.) would graphitize if subsequently held at 1200 degrees Fahr. (650 degrees Cent.). With respect to the types of carbides observed, it would also be interesting to know the maximum amount of molybdenum that can be taken into the orthorhombic lattice and the minimum amount of molybdenum in the face-centered cubic lattice.

It is stated that with increasing time at tempering temperature after quenching the amount of molybdenum in the carbide gradually increased, although at 250 hours it had not approached that obtained by direct transformation. Has the author, by any chance, obtained data for longer tempering times and would it be expected that the end product would be a face-centered cubic carbide of molybdenum content equal to that of the isothermal transformation product?

The writer was also interested in the author's remarks about an acicular structure which was observed on isothermal transformation at 1100 degrees Fahr. (595 degrees Cent.) and in which "the carbides were much more massive than those in the coarsest pearlite". We have observed a similar structure to form during the furnace-cooling, but not air-cooling, of certain 0.15 to 0.5 per cent molybdenum steels. Presumably during slow cooling, an opportunity is afforded for carbon segregation to occur to an extent sufficient to produce an austenite similar to that described by the author. It would be of interest if the author would present a photomicrograph of this structure.

In relation to the occurrence of both orthorhombic and face-centered cubic carbide on isothermal transformation, did the author discover any metallographic differences as to size or etching characteristics that would permit differentiation of the two carbide types?

The author's remarks on the relation of the partition data to hardenability were of great interest. The explanation of the effect of molybdenum in decreasing the rate of nucleation and growth which results in decreased overall reaction rate appears logical. The writer wishes, however, to point out that in connection with the occurrence of a temperature of maximum rate of transformation, above which carbide is probably the nucleating agent, it is not certain that it is ferrite, of the same sort that occurs at higher temperature, that nucleates the transformation at temperatures below the maximum, at least in plain carbon steels. Rather, there is good evidence to indicate that in the formation of these lower temperature products (bainite), a supersaturated sort of ferrite, much like martensite, appears first, and that carbide subsequently precipitates from this structure as suggested by Davenport and Bain.³ (See also footnote 4 for discussion of this point.) If this be true, the picture is not quite as the author has indicated, and evidently the maximum is brought about simply because the reaction to pearlite, which proceeds at a faster rate with lowered temperature, dies out, owing probably to an inability of forming a carbide nucleus necessary to initiate the reaction, and is replaced by another reaction which proceeds with decreased rate with lowered temperature.

The situation for alloy steels which show a "shelf" must be different as the author points out and explains by the statement that in plain carbon and low molybdenum steels the maximum rate of nucleation of carbide closely coincides with that of ferrite. It is difficult to understand the basis for this explanation unless it is assumed that the author has taken the coincidence of the ferrite and pearlite "beginning" lines of the isothermal transformation diagram at the region of maximum rate to mean this. But is this not reaction rate rather than nucleation rate? The author further postulates that in alloy steels which show a shelf in the isothermal transformation diagram, as in higher molybdenum steels, the maximum nucleation rate of carbide has been raised above that of ferrite thus resulting in a temperature range in which "austenite transformation is nucleated at a rate much slower than the maximum of either nucleating phase". Is not the author again fallaciously reasoning that the isothermal diagram is a plot of nucleation rate rather than reaction rate? In this connection must not the data of Hull, Colton and Mehl (author's ref. 13) represent rate of nucleation of pearlite and not simply of carbide since to form pearlite, carbide and ferrite must nucleate alternately? Further, if the author's explanation were correct it would be necessary to explain a maximum in carbide nucleation rate, for with still lowered temperature, the "beginning" curve of the pearlite reaction again moves to shorter times and coincides with the "beginning" curve of ferrite transformation at the lower temperature of maximum reaction rate. It appears to the writer that the explanation of the isothermal transformation curve of the type under discussion must be somewhat different than the author has indicated and probably is to be found in changes in the nature of the transformation products, structure and composition,

³E. S. Davenport and E. C. Bain, *Transactions, American Institute of Mining and Metallurgical Engineers*, Vol. 90, 1930, p. 117. See also subsequent papers from U. S. Steel Research Laboratory.

⁴G. V. Smith and R. F. Mehl, *Transactions, American Institute of Mining and Metallurgical Engineers*, Vol. 150, 1942, p. 211.

with changing temperature, and the attendant effects of these upon nucleation.

The author's data indicate that with lowered temperature there is less segregation of molybdenum. Would the author care to hazard a guess as to whether there would be any segregation at the temperature of maximum rate, which the writer presumes to be below the lowest temperature for which the author has obtained partition data? If not, there would appear to be some difficulty in explaining the increased hardenability of molybdenum (and presumably other) alloyed steels, since it is the rate of transformation at this temperature that primarily determines hardenability.

This paper is an interesting and worthwhile addition to the series of investigations being carried out in the author's laboratory on the subject of distribution of molybdenum in steels. It is to be hoped that these investigations will be continued and extended to hypo and hypereutectoid molybdenum steels and to other alloy steel systems, for it seems certain that the method of attack used by the author and his collaborators is one which will lead eventually to a more comprehensive understanding of the behavior of steels.

Written Discussion: By C. M. Offenhauer, research metallurgist, Union Carbide and Carbon Research Laboratories, Niagara Falls, N. Y.

This paper has been read with interest and we agree with the author that this type of investigation seems likely to result in a better understanding of the role of third elements in steels. One observation however, appears to require clarification. By comparing Tables I and II it may be seen that the same alloy, No. 38, showed the face-centered cubic structure of Mo_{23}C_6 when isothermally transformed at 1200 degrees Fahr. (650 degrees Cent.), and the orthorhombic structure of cementite when quenched and tempered at the same temperature. The alloys 71 and 72 also showed a difference in the carbide structure but this change was accompanied by some graphitization of the tempered specimens. In some investigations at the Union Carbide and Carbon Research Laboratories, we have found results similar to those reported by Mr. Bowman. Obviously, one of these carbides must be more stable than the other, even though both may eventually revert to graphite. We have postulated that in quenching to 1200 degrees Fahr. (650 degrees Cent.) the austenite might have undergone some change or decomposition before reaching 1200 degrees Fahr., and that the quenching and tempering process might permit the attainment of a more uniform structure at the temperature being investigated. In this case the orthorhombic structure would be considered the more stable phase at 1200 degrees Fahr. The range of stability of the face-centered structure appears to be at a higher temperature, and this is also indicated by the previous work of Bowman and Parke (5). In view of the persistent character of the alloy carbides, the method of determining the more stable phase is quite critical.

Oral Discussion

M. F. HAWKES:⁵ Anyone who has perused the literature to get information on why alloying elements in solution in austenite increase hardenability

⁵Instructor, Carnegie Institute of Technology, Pittsburgh.

will be very pleased to read this paper and Mr. Ham's, for the rest of the literature is certainly barren.

A question I have in mind applies to both papers, and perhaps both Mr. Bowman and Mr. Ham would like to discuss it. It is hard for me to see why it is necessary for alloying elements to be partitioned to a definite degree between ferrite and carbide. Is it not possible that alloying elements affect the rate of transformation of austenite in some other manner and that the amount of partition occurring is merely the amount determined by the time available and the rate of diffusion of the alloying element? This is in agreement with the observation that the degree of partitioning decreases as the reaction temperature decreases.

Author's Reply

I wish to express my appreciation to Mr. Smith, Mr. Offenhauer, and Mr. Hawkes for their comments.

In reply to Mr. Smith's remarks on graphitization, it seems to me that the stability of the carbides is related more closely to their composition and structure than to a nucleation effect. Smith, Miller, and Tarr^a have shown, by converting graphite to carbide at 1300 degrees Fahr. (705 degrees Cent.) in a low-carbon molybdenum steel, that at this temperature the carbide is the stable phase. No identification of the structure of this reconverted carbide was attempted; however, steels of similar analysis yielded the face-centered cubic carbide after isothermal transformation at 1300 degrees Fahr. (705 degrees Cent.), and it would be surprising if the carbides found by Smith were not of the same type.

No data have been obtained for tempering times greater than 250 hours at any temperature; consequently, no definite statement can be made about the end product of the tempering treatment. Nevertheless, since it is felt that the results of isothermal transformation approximate equilibrium conditions, the results of tempering martensite should eventually correspond to those obtained by direct transformation. It is possible, of course, that graphite may be formed as an intermediate phase.

No additional data are available on the maximum molybdenum content of the orthorhombic carbide or the minimum molybdenum content of the face-centered cubic carbide. Data already reported indicate that about 3.5 per cent, by weight, molybdenum is necessary in the carbide to cause an alteration in structure. This has been substantiated by Dr. F. R. Morral in a private communication wherein he pointed out that the minimum molybdenum content of the face-centered cubic carbide may be represented by the formula $(\text{Fe}_{23/4}\text{Mo}_{1/4})\text{C}_6$. I have found no evidence that the orthorhombic carbide exists with a molybdenum content greater than this.

It is quite possible, as Mr. Smith points out, that the ferrite formed during the transformation to bainite is not originally the usual low-carbon alpha iron but a supersaturated solution, but it does not seem to me that this in any way invalidates the explanation proposed for the effect of molybdenum on hardenability. Judging from the isothermal diagrams, the effect of molybdenum, as

^aG. V. Smith, R. F. Miller and C. O. Tarr, *Metals Technology*, June 1944, Technical Publication No. 1695.

well as of most other alloying elements, is restricted to the region where the reaction is nucleated by carbide. It is immaterial, as far as the proposed explanation is concerned, just how the bainite reaction is initiated. The important fact is that the nature of the transformation is altered.

I am fully aware that the "beginning" lines of isothermal transformation diagrams are not plots of rates of nucleation; however, they serve as a rough approximation of the time required to form nuclei. It is true that the data of Hull, Colton and Mehl⁷ represent the rate of nucleation of pearlite and were obtained from the number of pearlite colonies of observable size formed in a given time; but, since each colony—according to present theories—is nucleated by a carbide, is not this rate an indication of the carbide nucleation rate?

Mr. Smith's objection to the explanation of the "shelf" in the isothermal transformation curves of the higher molybdenum alloys is based upon the necessity for explaining a second maximum in the rate of carbide nucleation. I do not believe that this second maximum exists. Metallographic evidence already published (2) reveals that the products of transformation at temperatures below the second minimum in the transformation are of the bainite type. There are no data available which obviate the idea that both the pearlite and the bainite reaction curves are essentially "C" curves or that their location with respect to temperature determines the general shape of the over-all "S" curves.

Although it has been impossible to obtain partition data from alloys transformed at the maximum rate, it would be surprising if some segregation of molybdenum in the carbide had not occurred.

Insufficient data are available to answer Mr. Offenhauer's remarks concerning the relative stability of the two carbides. The results thus far obtained, however, indicate that the conditions existing after isothermal transformation more closely represent the equilibrium conditions at the transformation temperature than do those obtained by tempering martensite at this temperature for the length of time reported in the paper. The results of the latter treatment indicate a gradual increase in the molybdenum content of the carbide with time, yet in the time intervals investigated this molybdenum content did not reach that obtained by direct transformation. It would be strange if the ultimate equilibrium conditions were different when approached from different directions.

Mr. Hawkes' point about the possibility that partition is more a result than a cause is well taken. It is impossible to make quantitative statements regarding the determining effect of available time and diffusion rates upon partition without an accurate knowledge of the diffusion distances involved. After having been only a few seconds at the transformation temperature, the partially transformed alloys contain the high-molybdenum, face-centered carbide, yet after tempering martensite for two hundred and fifty hours the same alloy still contained the orthorhombic carbide. In view of these facts it seems that the partition of molybdenum is actually a part of the transformation process and exerts an effect upon it.

⁷F. C. Hull, R. A. Colton, and R. F. Mehl, *Metals Technology*, August 1942, Technical Publication No. 1460

NEW DEVELOPMENTS IN HIGH STRENGTH ALUMINUM ALLOY PRODUCTS

By E. H. DIX, JR.

Abstract

Until recently, aircraft structural aluminum alloy products, exclusive of castings and forgings, have been used in the "naturally aged" temper. Higher strengths can be obtained only by "artificial aging," i.e., elevated temperature precipitation treatment subsequent to solution heat treatment, but this treatment results in a high ratio of yield to ultimate strength, a lower elongation and sometimes a lower resistance to corrosion. These latter factors have retarded the general acceptance of products so treated. However, changes in design trends and in engineering viewpoint have resulted in overcoming previous objections and placing special emphasis on high compressive yield strength.

The highest compressive yield strengths are now obtainable in products of three artificially aged aluminum alloys—24S-T8, 14S-T and 75S-T. The mechanical properties of artificially aged 24S depend upon the amount of cold working after the solution heat treatment and before the elevated temperature precipitation treatment. The highest available yield strength in an aluminum alloy sheet product is obtained in 24S-T86 by cold working approximately 5 per cent after solution heat treatment and then artificially aging. The alloy 14S-T has the shop advantage of a relatively stable and workable room temperature aged temper, permitting parts to be formed and subsequently artificially aged without distortion. Extrusions of the aluminum-zinc-magnesium type alloy, 75S-T, have the highest yield and tensile strengths, and sheet the highest tensile strength combined with high yield strength and good elongation.

THE designer of aircraft structures today seems to be primarily interested in the compressive yield strength of the new high strength aluminum alloy products. This is somewhat in contrast to

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, E. H. Dix, Jr., is assistant director of research and chief metallurgist, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa. Manuscript received June 21, 1944.

the attitude a few years ago. The increase in size and weight of aircraft, the trend toward thinner air foils requiring the use of thicker sections, more exact design data and war demands are probably largely responsible for this change. Furthermore, there has been a gradual evolution in engineering viewpoint regarding the mechanical characteristics which are required in a structural material suitable for aircraft. Formerly, more emphasis was placed on the necessity of a generous spread between yield and tensile strengths, on elongation, on impact strength, and on resistance to corrosion. Aircraft structural engineers still differ widely as to the importance which they attach to these properties. Even now, tensile yield strengths exceeding approximately two-thirds of the tensile strength cannot be taken advantage of in the design of tension members.

The new high strength aluminum alloy products all achieve their maximum strength by artificial aging. Artificial aging increases the yield strength to a much greater degree than it does the tensile strength. Thus the ratio of yield-to-tensile exceeds the two-thirds value and therefore the high tensile yield strength generally cannot be utilized in members designed for tension loads. In the artificially aged products the compressive yield strength is approximately equal to the tensile yield strength and therefore these products are admirably suited for compression members, especially for effective slenderness ratios of less than about 40.

Artificial aging is a term long used in the aluminum industry to describe a reheating treatment subsequent to the high temperature solution heat treatment. The name apparently originated because the reheating was thought to stimulate or artificially produce, in a short time, aging similar to that which occurs at room temperature over longer periods of time. More correctly, it should be described as an elevated temperature precipitation treatment since long time aging tests at room temperature do not support the original opinion.

The response of certain aluminum alloys to artificial aging was described by Archer and Jeffries (1).¹ These authors described the effect of added silicon in increasing the response of the duralumin type alloy (17S) to artificial aging. They thus laid the groundwork for the development of artificially aged duralumin-type alloys.

These were two in number—14S and C17S. Later foreign publications sometimes refer to alloys of this type as super-duralumin (2), (3), (4). The nominal compositions of these two alloys

¹The figures appearing in parentheses pertain to the references appended to this paper.

are quite similar. As will be seen from the following tabulation, 14S contains somewhat higher copper and manganese but lower silicon and magnesium than C17S.

	Per Cent			
	Copper	Magnesium	Manganese	Silicon
14S	4.4	0.35	0.75	0.80
C17S	4.0	0.50	0.50	1.25

The nominal magnesium content of 14S was subsequently raised to 0.40 per cent.

Data (5) published as early as 1927 listed Special 17S (later designated "C17S") sheet with a tensile strength of 63,000 to 70,000 pounds per square inch, a yield strength of 50,000 to 55,000 pounds per square inch, and an elongation of 8 to 14 per cent in the artificially aged temper (T). An advantage of the alloy was that it could be formed in the W, or room temperature aged, temper and subsequently artificially aged without appreciable distortion. The effect of the artificial aging in reducing the resistance to corrosion of this sheet material was also observed.

This sheet product was not favorably received, probably because of the prevailing engineering viewpoint concerning the mechanical characteristics of structural materials. Thus, the high yield strength in relation to tensile strength, and the relatively low elongation were questioned. The lower resistance to corrosion of this product, in comparison to the currently used aluminum alloy sheet (17S-T) is undoubtedly another reason. The latter objection was overcome to some extent by the experimental production of Alclad C17S-T sheet. For instance, Alclad C17S-T sheet was used for girder members for a trapeze on the U.S.S. Los Angeles, a rigid dirigible airship, in 1931 and showed no significant corrosion when examined eight years later. However, the production of C17S-T sheet was discontinued prior to 1935 because of lack of customer acceptance.

The development of 14S for forgings paralleled somewhat that of the C17S sheet. It was first listed (6) in 1932 but had been used experimentally considerably prior to that date. The status of the alloy for forgings in 1932 was that it was recommended where highest strengths were required, provided the part was not too complicated.

The acceptance of the 14S-T composition for forgings was in marked contrast to the experience with sheet. Today it is still the principal aluminum alloy for high strength aircraft forgings and

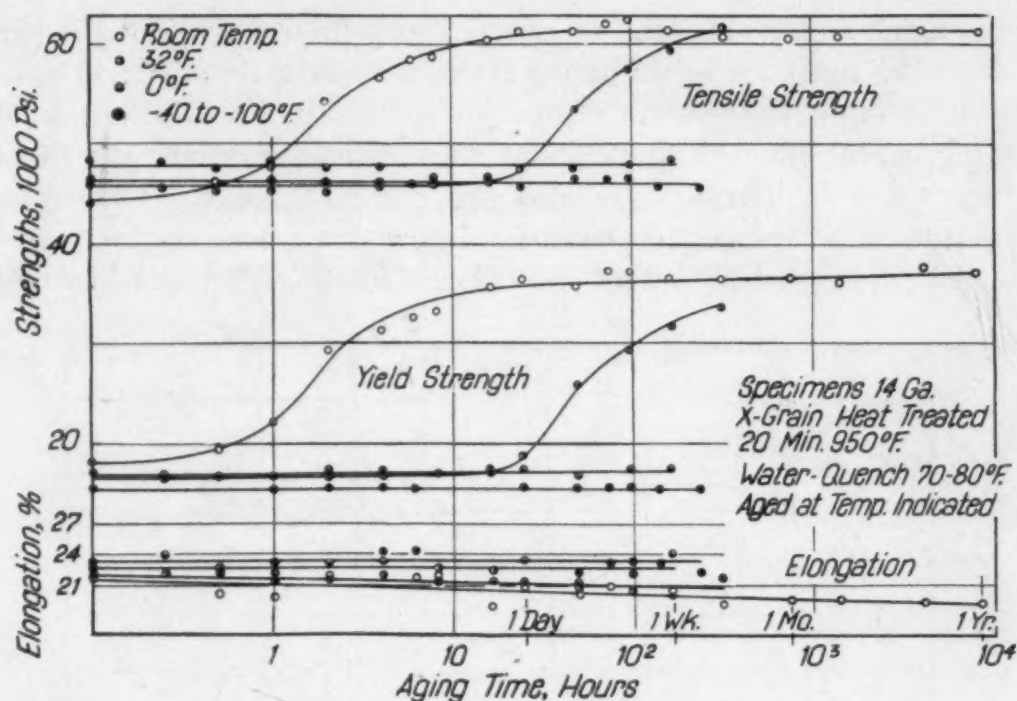


Fig. 1—Natural Aging of 17S at Room Temperature and Below.

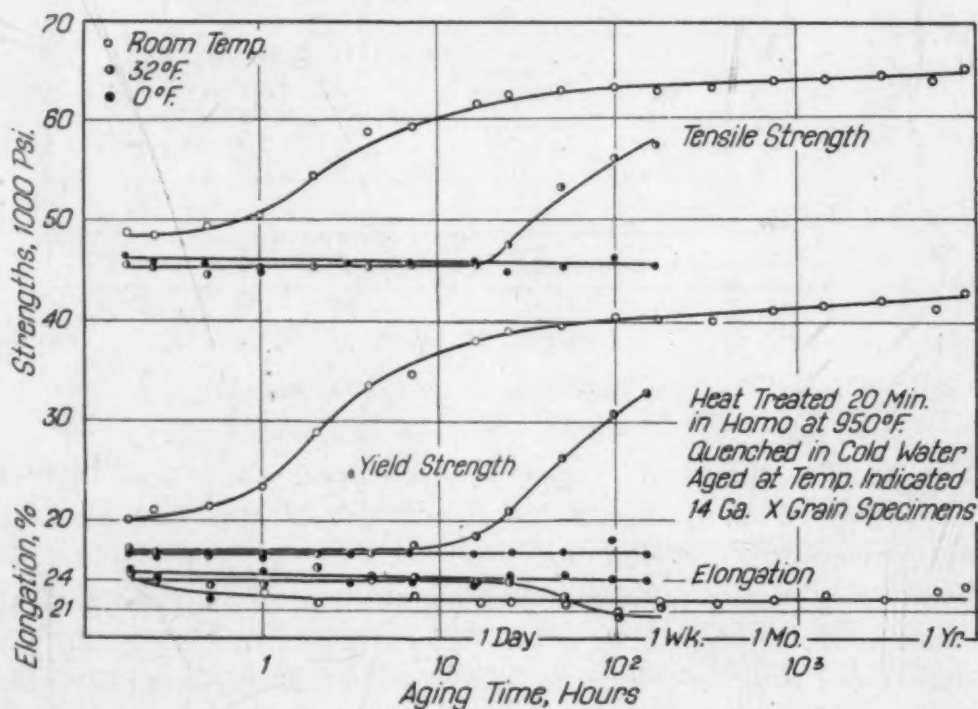


Fig. 2—Natural Aging of 14S at Room Temperature and Below.

many millions of pounds have given excellent service in peace and war.

It will be of interest to compare the "natural" or room tempera-

ture aging and the artificial or elevated temperature aging of the two alloys 17S and 14S, remembering that the essential difference in composition is the high silicon content of the latter alloy. The natural aging curves for 17S and 14S at room temperature are shown in Figs. 1 and 2. These graphs also show the retarding effect on aging of storage at several temperatures below room temperature. Retarded aging is of advantage for difficult forming operations carried

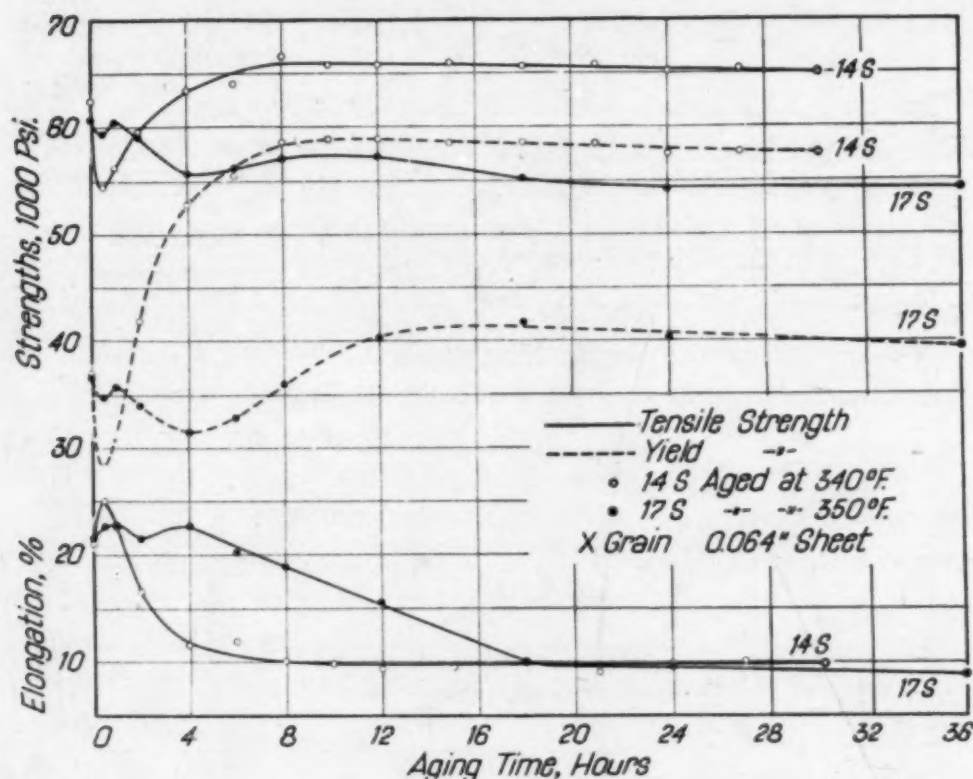


Fig. 3—Artificial Aging of 17S and 14S Sheet.

out in the "as-quenched" temper. The curves show that there is little difference in the aging characteristics of these two alloys at room temperature or below.

The response to an elevated temperature precipitation treatment or an artificial aging of these two alloys is shown in Fig. 3. Although there is some slight increase in yield strength in 17S, this increase is more than offset by the decrease in tensile strength and elongation. On the other hand, after an initial dip in tensile and yield strengths, the yield strength of 14S is increased 50 per cent by artificial aging. The tensile strength is increased perhaps 5000 pounds per square inch and the elongation about cut in half. The

ratio of yield to tensile strength is increased from approximately 0.6 to approximately 0.9.

Fig. 4 shows a series of artificial aging curves illustrating the effect of time and temperature in the artificial aging treatment. These curves are fairly typical of artificial aging curves in general

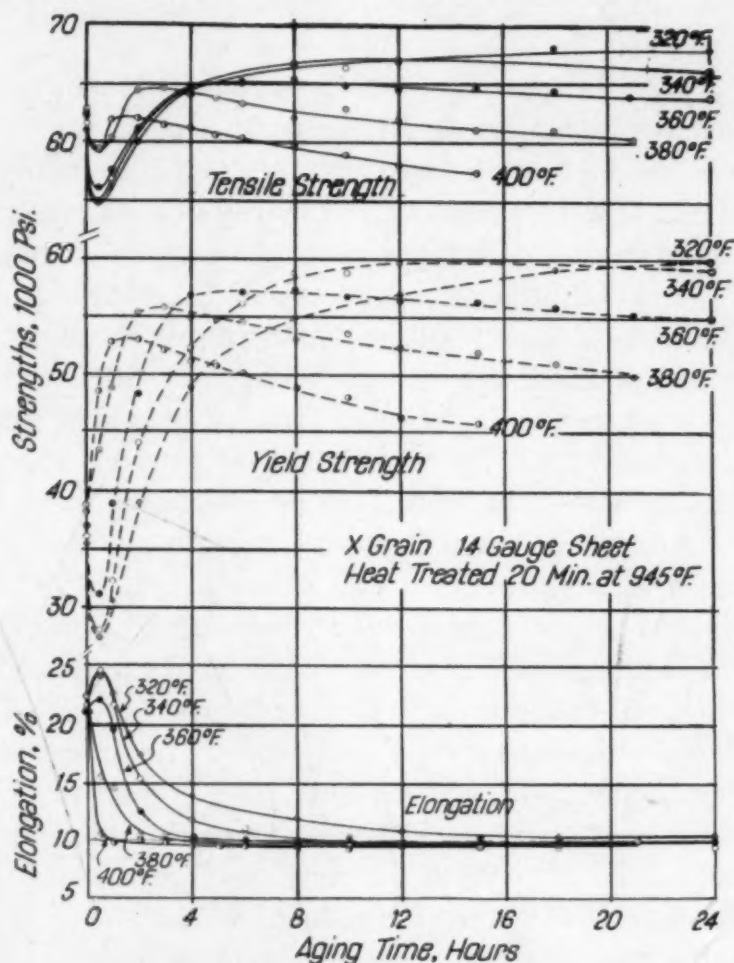


Fig. 4—Artificial Aging Curves for 14S Sheet. Heat Treated 20 Minutes at 945 Degrees Fahr. and Cold-Water-Quenched.

but it is probable that no two lots of similar alloy composition will age along identical curves. It will be observed that as the temperature of artificial aging is increased, the time for reaching the peak of tensile and yield strengths is reduced, the curves become more abrupt, and, in general, the maximum values of tensile and yield strengths are somewhat lower. In selecting the time and temperature of artificial aging, a compromise must be reached between the desire, on the one hand, for keeping the furnace time to a minimum and, on the other, for selecting a relatively flat curve so that varia-

tions in the aging time resulting from differences in the rate of heating in different parts of the load will have a relatively minor effect. Thus, for 14S, 10 hours at 340 degrees Fahr. (170 degrees Cent.) is probably the most satisfactory selection, with other treatments of 18 hours at 320 degrees Fahr. (160 degrees Cent.) or 5 hours at 360 degrees Fahr. (180 degrees Cent.) producing approximately equivalent results.

Since 1932 (7), the alloy 24S and Alclad 24S have been universally used for highly stressed structural parts of current aircraft. This alloy is the highest strength naturally aged aluminum alloy in commercial use. It differs from 17S primarily in a magnesium content of 1.5 per cent instead of 0.5 per cent. During the development of 24S it was found that artificial aging reduced the elongation to a value too low to be acceptable at that time. Hence, until recently, it was used in the room temperature aged temper only. For this alloy the room temperature aged temper is designated "T". It is also furnished in a temper designated "RT", produced by cold working approximately 5 to 6 per cent subsequent to the solution heat treatment and room temperature aging. The tensile yield strength of this product is greater than the compressive yield strength. In 1939 the Aluminum Company co-operated with an American builder of dirigibles to determine if, as suggested in an article by Brenner and Kostron (8), an elevated temperature aging treatment of 24S-RT sheet would raise the compressive yield strength. A treatment of 8 hours at 350 degrees Fahr. (175 degrees Cent.) was found to increase the compressive as well as the tensile yield strength and to make them approximately equal but only at the sacrifice of resistance to corrosion and elongation. However, in the case of alclad sheet, it was concluded that the resistance to corrosion would be adequate. The decision, however, was not to employ this artificially aged material for dirigible structures. Probably the low elongation was the deciding factor.

Others experimented with the artificial aging of 24S products but it was not until within the past couple of years when several of the aircraft manufacturers (9), (10) on the west coast became interested in the possibility of artificially aging these products, and indicated a willingness to accept the lowered elongation. It was then found that not only could the properties of the cold-worked tempers of this alloy be improved by artificial aging but also that the small amount of cold deformation introduced by mill flattening had a pro-

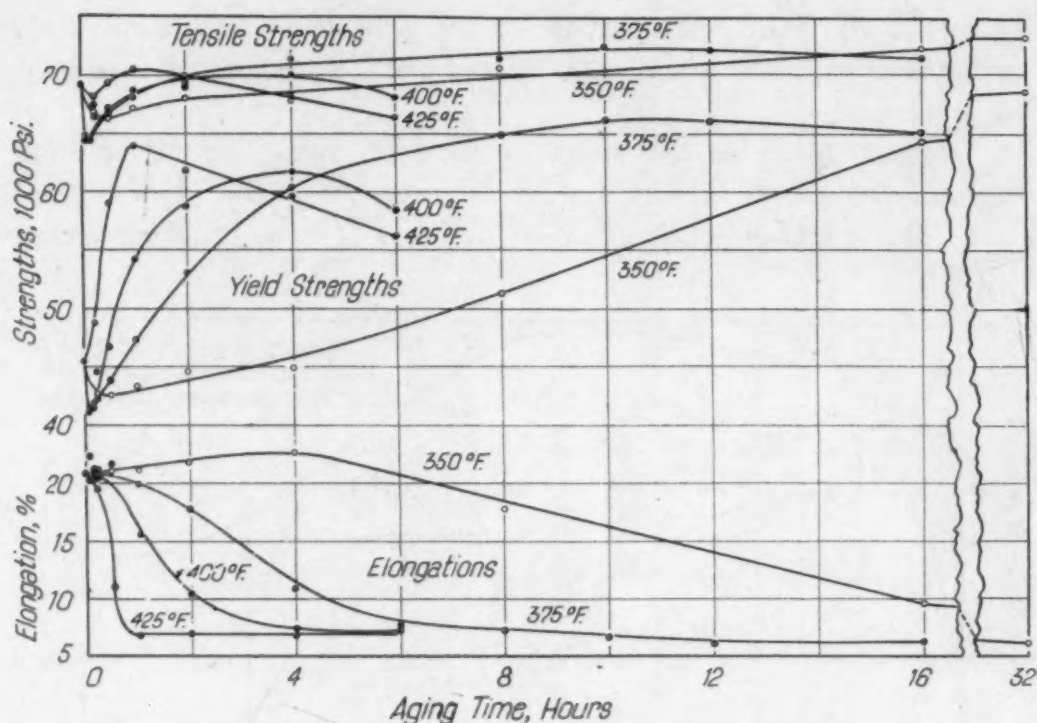


Fig. 5—Artificial Aging Curves for 24S-T Flat Sheet.

nounced effect and that, even without any cold work, some improvement could be achieved. The response of 24S-T flat sheet to artificial aging treatments is shown in Fig. 5. It will be observed that 10 to 12 hours at 375 degrees Fahr. (190 degrees Cent.) gives maximum tensile and yield strengths for 24S-T flat sheet. The marked reduction in elongation from an average of about 21 to 6 or 7 per cent is notable as is also the increase in ratio of yield strength to tensile strength from about 0.65 to over 0.90.

Detailed studies of the effect of artificial aging on resistance to corrosion yielded the important information that although short reheating times had a marked detrimental effect on resistance to corrosion, if the time is prolonged until maximum yield and tensile strengths are obtained, the resistance to corrosion may be restored to that of the original T temper. Fig. 6 shows the per cent loss in tensile strength caused by a standard accelerated corrosion test of sheet specimens $\frac{1}{16}$ inch thick which had been artificially aged for different periods of time at several different temperatures. Thus, at a temperature of 375 degrees Fahr. (190 degrees Cent.), heating for 1 hour produces a material which lost 50 per cent in tensile strength as compared to 17 per cent in the original temper. However, heating

for a total of 10 to 12 hours at which the peak in the tensile and yield strengths is obtained practically reduced the loss to that of the original T temper.

Measurements of the effect of artificial aging on the solution potential are shown in Fig. 7. At 375 degrees Fahr. (190 degrees Cent.) the solution potential in 10 to 12 hours has reached almost

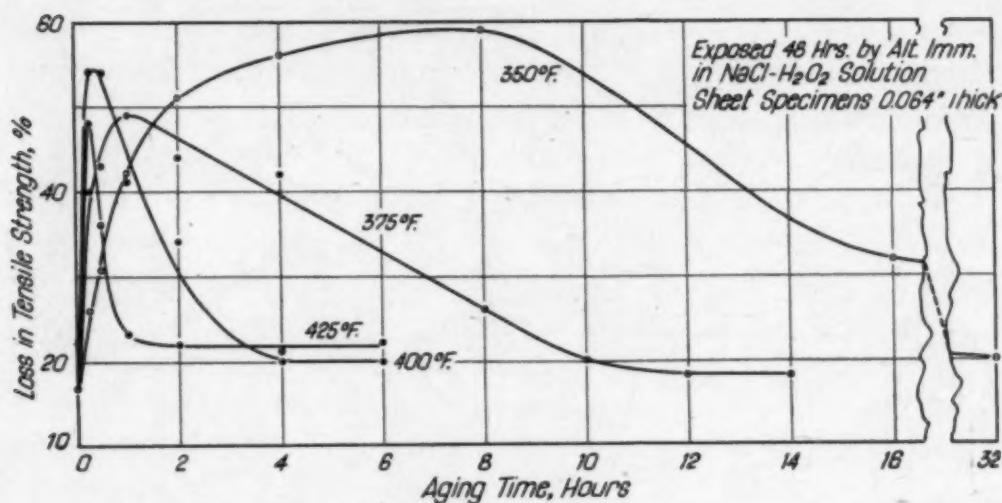


Fig. 6—Effect of Artificial Aging on Resistance to Corrosion of 24S-T81 Under Stress of Three-Quarters of the Yield Strength.

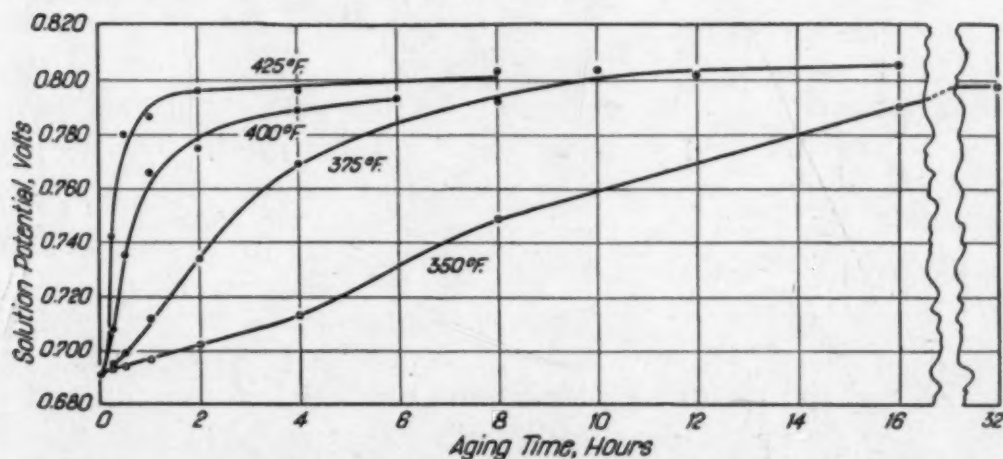


Fig. 7—Solution Potential of 24S-T Flat Sheet as Affected by Artificial Aging.

its maximum value indicating the almost complete precipitation of copper from solid solution. There is good evidence now to substantiate the belief that the short heating period at 375 degrees Fahr. (190 degrees Cent.) produced precipitation which was primarily localized at the grain boundaries, thereby causing marked susceptibility to intergranular corrosion. More extended heating, however, caused

general precipitation within the grains and thereby equalized the potential at the grain boundaries and within the grains, thus removing the cause of selective corrosion.

With alclad sheet, although the resistance to corrosion of the core may be substantially restored to that of the as-quenched material by the use of proper time and temperature, yet, as will be seen from Fig. 7, the solution potential of the core is raised to almost that of the pure aluminum alclad coating. In the absence of copper

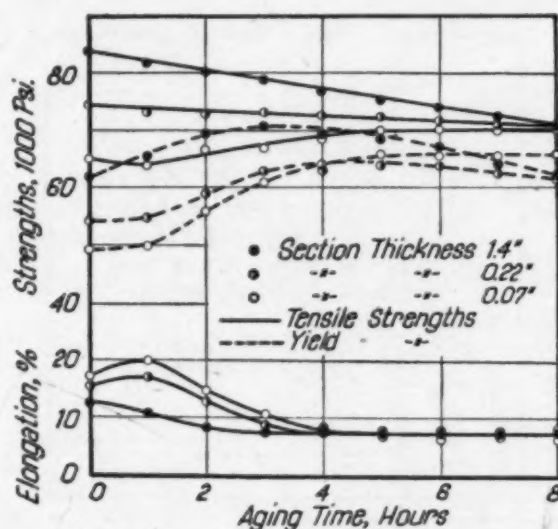


Fig. 8—Effect of Section Thickness Upon Longitudinal Properties of 24S-T8 Extrusions Aged at 390 Degrees Fahr.

diffusion from the core, the solution potential of the coating is 0.83 volt. The presence of copper diffusion causes a lowering of this value. The alclad coating is still effective as a mechanical protection and the resistance to corrosion of the product, except for very thin sheet, is adequate.

With thin alclad sheet or with thicker sheet of reduced coating thickness, the diffusion of copper from the core into the coating may be sufficient that, after artificial aging, the coating itself may be susceptible to intergranular corrosion and the resistance to corrosion of the product such that careful protection methods are necessary.

The response of 24S-T extrusions to artificial aging is a somewhat more complicated story. It is well known and recognized in Government specifications that the longitudinal tensile and yield strengths of 24S-T extrusions vary with the section thickness. The thicker the section, the higher the strengths. The points for zero

aging time in Fig. 8 illustrate the influence of section thickness on the longitudinal properties of the 24S-T extrusions. The effect of artificial aging at 390 degrees Fahr. (200 degrees Cent.) for different periods of time on these three section thicknesses, as shown in Fig. 8, is of considerable interest. The thinner section shown by the open circles behaves somewhat analogous to sheet in that the yield and tensile strengths are increased by the artificial aging, maximum values being obtained after about 6 hours at this temperature. With the intermediate thickness, the yield strength is also increased

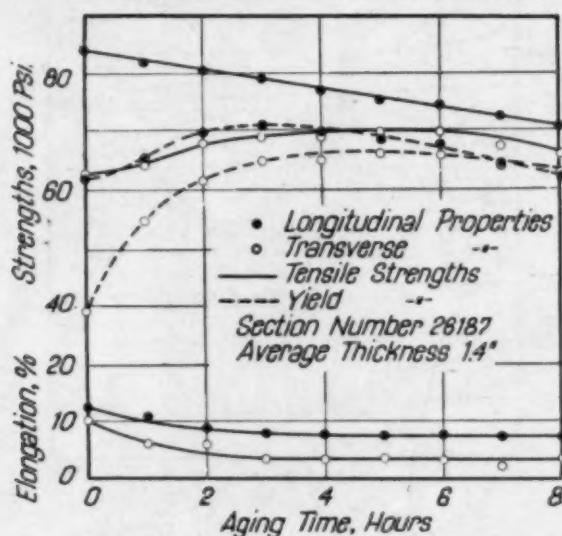


Fig. 9—Longitudinal and Transverse Properties of 24S-T8 Extruded Section Aged at 390 Degrees Fahr.

somewhat although the peak is reached in about 4 to 5 hours. The tensile strength shows a slightly downward trend. With the heavier section, however, the decrease in tensile strength is quite noticeable and the gain in yield strength smaller with the peak at about 3 hours of heating. In Fig. 9 the curves for the longitudinal properties of the thickest extrusion are reproduced together with the transverse properties of this extrusion. First it should be observed that transverse properties as indicated for zero aging time are very much lower than the longitudinal properties. This is characteristic and well known. The artificial aging produces a marked increase in the transverse yield strength and some increase in the transverse tensile strength. The peak of the transverse tensile strength is obtained after about 3 hours of aging, whereas the maximum for the transverse yield strength is fairly constant in 4 to 6 hours and is practically equal to the longitudinal yield strength.

From the typical results shown in Figs. 8 and 9, the artificial aging of large 24S-T extrusions is not generally beneficial and there is some question if it will be used except in special cases and possibly with thin sections.

The information on the effect of artificial aging of aluminum alloys on their fatigue characteristics is not great. In general, however, it may be said that artificial aging has very little effect on the endurance limit. Very limited data suggest that the notch sensi-

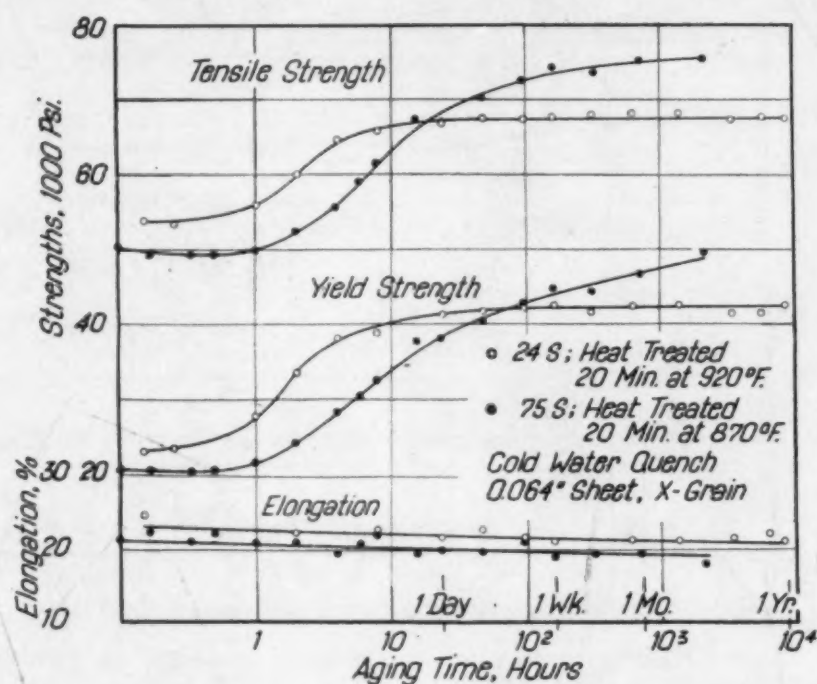


Fig. 10—Room Temperature Aging of 24S-T and 75S-T Sheet.

tivity in fatigue may be increased somewhat. Until more data are available it will probably be wise to give more consideration to the fatigue strength of structures utilizing these high strength artificially aged alloys than has been necessary with room temperature aged products.

The latest addition to the Alcoa family of alloys is 75S. This is now available as alclad sheet and extrusions. Its principal hardening elements are zinc and magnesium, with smaller additions of copper and several other elements. It represents the culmination of over 15 years of laboratory and manufacturing research and development. It is the present ultimate in high strength aluminum alloy development.

The combination of zinc and magnesium as a hardening element

in heat treated aluminum alloys was described by Sander and Meissner in 1926 (11). Exploitation of alloys of this type in Germany under the trade name of "Constructal" was doomed to failure because of their marked susceptibility to stress corrosion cracking. About 1937 the British Air Ministry issued Specification DTD 363 covering broadly an alloy of this general type, known as "RR 77". This alloy also encountered the stress corrosion cracking snag in extrusions which were cold-deformed after heat treatment and its

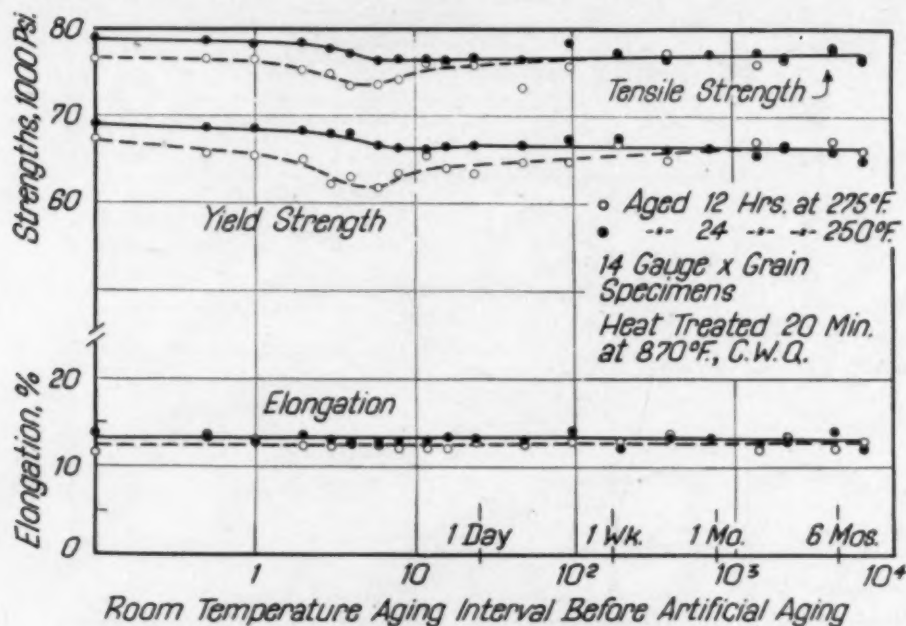


Fig. 11—Effect of Interval Between Quenching and Aging on Mechanical Properties of Alclad 75S-T Sheet.

use has been rather limited. By 1932 (12), there had been developed in this country an experimental alloy which, in the artificially aged temper, had a yield strength in excess of 80,000 pounds per square inch but its other characteristics were not satisfactory for general structural applications. However, by 1939 research achieved sufficient progress towards the ultimate goal as to justify presenting the facts to the military authorities of the United States in order to enlist their interest.

Co-operative experiments with the Government and several aircraft manufacturers indicated that further improvement in resistance to stress corrosion cracking was desirable. During the next three or four years such improvement was obtained and in the fall of 1943 the first large production lot of 75S was produced.

The alloy 75S also requires artificial aging to reach its maximum

strength. It probably will be used commercially only in the T temper. Immediately after quenching and for an hour or two thereafter at room temperature, its yield strength is not appreciably higher than that of the annealed material. In the T temper its formability is definitely lower than that of 24S-T. Therefore the optimum method of producing formed parts will be in the soft condition after quench-

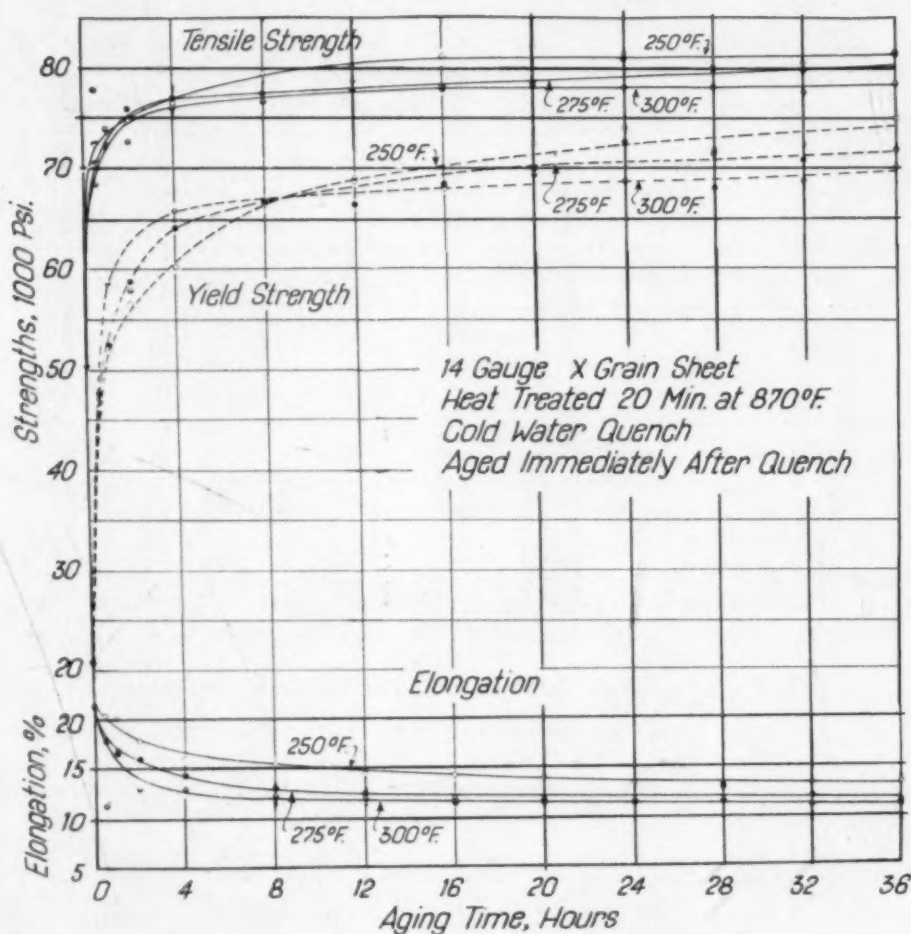


Fig. 12—Artificial Aging Curves for 75S-T Sheet Aged Immediately After Quenching.

ing. Fig. 10 compares the room temperature aging characteristics of as-quenched 24S and 75S and it will be observed that up to approximately one day, the yield and tensile strengths of 75S are lower than those of 24S. By resorting to storage at refrigeration temperatures, the aging of 75S as well as 24S can be prevented for any desired period, thus avoiding the necessity of close scheduling between heat treatment and forming.

The aging of 75S, like that of 14S, is not affected by cold work between the solution heat treatment and the artificial aging treat-

ment. However, the time interval between quenching and the beginning of artificial aging does influence the final properties obtained. This is shown in the curves of Fig. 11. The highest final properties are obtained when artificial aging is started, within 2 hours after

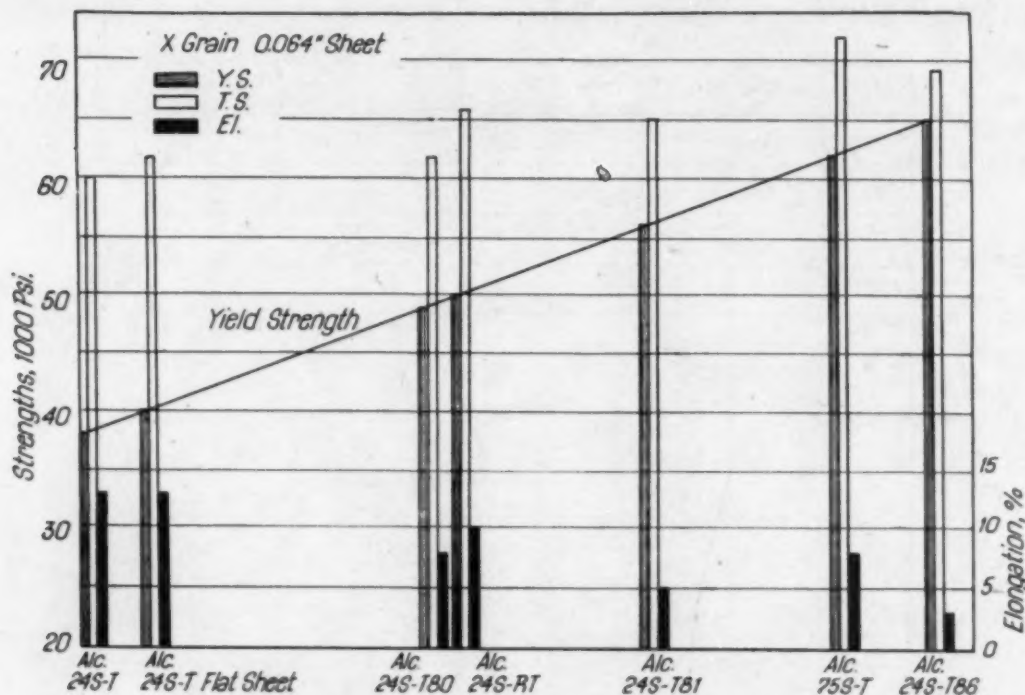


Fig. 13—High Strength Aluminum Alloy Alclad Sheet (Minimum Specification Values).

quenching, or after a room temperature aging period of a minimum of 2 days.

The artificial aging characteristics of 75S at three different temperatures are shown in Fig. 12. It will be observed that the best combination of properties is obtained after aging for 24 hours at 250 degrees Fahr. (120 degrees Cent.). 75S extrusions respond to artificial aging in a similar manner to sheet.

The alloy coating on Alclad 75S has been selected so that it exercises electrolytic protection over the core even in the artificially aged temper. Thus, the resistance to corrosion and stress corrosion cracking of Alclad 75S-T sheet is excellent. It is the consensus of those who have studied the results of accelerated tests and limited atmospheric exposures that if 75S-T extrusions are treated according to the procedures found desirable for 24S-T extrusions they will give satisfactory service in military aircraft.

Alloys of the 75S type have an endurance limit in the range of 20,000 to 22,000 pounds per square inch as compared to 18,000 pounds per square inch for 24S-T. In this respect 75S-T extrusions are notably superior to the duralumin-type alloys. The notch sen-

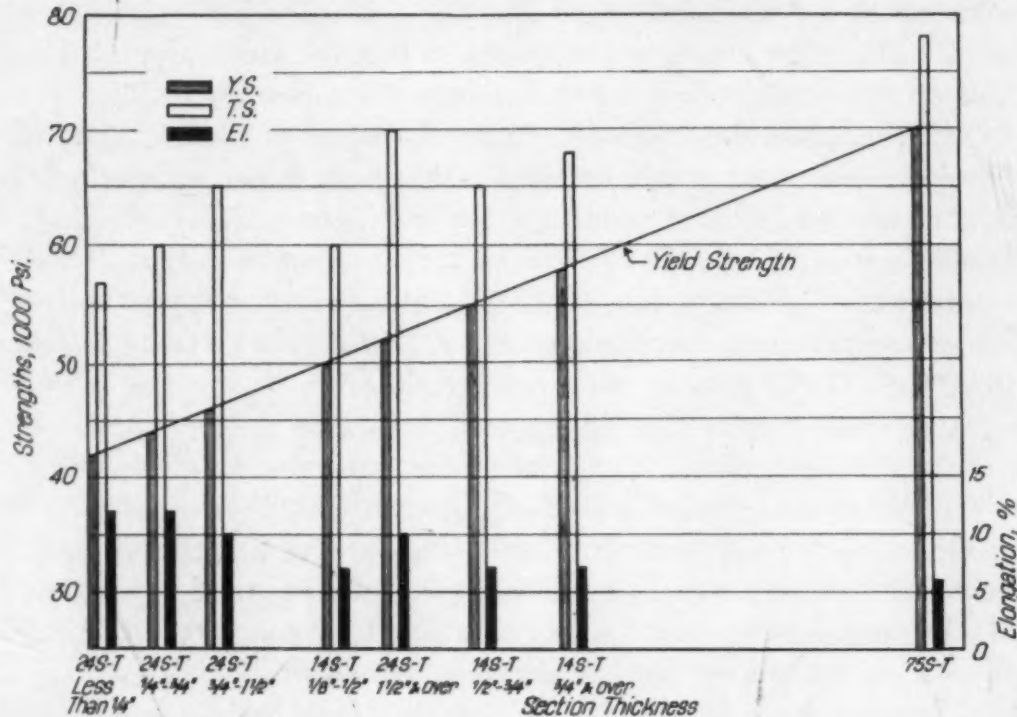


Fig. 14—High Strength Aluminum Alloy Extrusions (Minimum Specification Values).

sitivity in fatigue, however, may be somewhat greater than for the latter alloys.

The mechanical properties* of the new high strength aluminum alloy products are shown graphically in Fig. 13 (for alclad sheet) and Fig. 14 (for extrusions). These properties are Government spec-

*The first duralumin-type wrought structural alloy produced in this country (17S) was an alloy susceptible only to room temperature aging. The letter "T" was selected to represent the heat treated condition of this product, i.e., solution heat treated and room temperature aged at least 4 days. When other alloys, such as 14S, were developed, which were used in both the room temperature aged and artificially aged tempers, the letter T was retained to designate the fully heat treated condition, that is, the temper after artificial aging. The letter W, then, was instituted to designate the room temperature aged temper. Common usage now employs W to indicate as-quenched material. When so used, the condition is indefinite unless the period of room temperature aging is also stated. However, where mechanical property minima for this temper are given in specifications a minimum aging time of 4 days is assumed.

The room temperature aged temper of 24S is designated as T. The symbol T8 is used generically to indicate solution heat treatment followed by cold work followed by elevated temperature precipitation. A second numeral is used to indicate roughly the degree of cold working imposed between solution heat treatment and artificial aging. Thus, T80, designating artificially aged unflattened sheet, may be remembered because the 0 indicates substantially no cold working between solution heat treatment and artificial aging. T81 designates commercial flat sheet artificially aged, the 1 being remembered as indicating roughly about 1 per cent cold work introduced by the flattening. T84 is the temper designating roll formed sections which are stretched 3.5 per cent to 4 per cent after solution heat treatment and then artificially aged. T86 indicates the artificially aged temper of RT sheet which has received approximately 5 to 6 per cent cold work prior to artificial aging.

ification minima. The products are arranged in order of increasing yield strength. Thus, for Fig. 13, a yield strength of 38,000 pounds per square inch for Alclad 24S-T coiled strip or other unflattened sheet is shown at the left and a yield strength of 65,000 pounds per square inch for artificially aged 24S-RT (24S-T86) is shown at the right. The other products are spaced so that the yield strength block falls on the straight line drawn between these two points.

The effect of the cold work during flattening of the Alclad 24S-T flat sheet is shown by an increase of 2000 pounds per square inch in yield strength. The next product is the unflattened Alclad 24S-T artificially aged (24S-T80). Following this product is Alclad 24S-RT in which approximately 5 to 6 per cent cold reduction after the solution heat treatment has increased the tensile yield strength from 38,000 to 50,000 pounds per square inch, with an increase also of 4000 to 5000 pounds per square inch in tensile strength and some reduction in elongation. The next product in order of yield strength is artificially aged Alclad 24S-T flat sheet (Alclad 24S-T81). The small amount of cold working during the flattening operation has so increased the response to artificial aging that a yield strength of 56,000 pounds per square inch is obtained as compared to 49,000 pounds per square inch in the absence of cold work.

Next in order is Alclad 75S-T, with a yield strength of 62,000 pounds per square inch and the highest tensile strength (72,000 pounds per square inch) of any of the new products. Thus the ratio of yield-to-tensile strength is more favorable. Also the elongation is relatively high. The product with the highest yield strength is the artificially aged Alclad 24S-RT sheet (Alclad 24S-T86) but the ratio of yield-to-tensile is very high and the elongation low.

In all of the products listed, except Alclad 24S-RT sheet, the compressive yield strength is approximately equal to the tensile yield strength. Hence the graphs in Fig. 13 show the designer of aircraft structures the relative weight saving which may be accomplished by the use of these products. To achieve the ultimate he would choose between Alclad 75S-T and Alclad 24S-T86 depending upon the importance that he would attach to the ratio of yield-to-tensile strength and elongation. However, in order to use the yield strength of Alclad 24S-T86 only a very limited amount of forming is permissible because the part must be formed of Alclad 24S-RT and then artificially aged. Likewise, the properties of Alclad 24S-T81 can only be achieved for those parts which can be formed from Alclad 24S-T

flat sheet. The properties of Alclad 24S-T80 and Alclad 75S-T can be obtained from parts formed in the annealed temper, the as-quenched temper or the T temper, since these properties are not dependent upon cold working after heat treatment. Hence, for maximum compressive yield strength of parts requiring any except the simplest forming operations the choice would seem to fall on Alclad 75S-T.

The sheet products R301 and Alclad 14S, alclad sheet having a core of 14S and a copper-free magnesium-silicide type coating, have properties which would indicate that these products would fall in about the same position in the chart as Alclad 24S-T81. They would have the advantage over the latter product, however, in that because of the characteristics of the 14S core they could be formed in the as-quenched or W temper and then artificially aged to full strength; that is, this product also would not require cold work subsequent to the solution heat treatment in order to achieve the full response to artificial aging.

Alclad 24S-T84 is a special product produced as roll formed sections which are stretched 3.5 to 4 per cent after the solution heat treatment and subsequently aged. It is not included in the graph since, by the nature of the product, no transverse properties have been established. The minimum specification longitudinal values are tensile strength 70,000 pounds per square inch, yield strength 66,000 pounds per square inch, elongation 5 per cent.

The graphs for extrusions in Fig. 14 are arranged in a manner similar to that described for Fig. 13. The effect of section thickness on the properties of 24S-T and 14S-T extrusions are illustrated. The exceptionally high properties obtained in 75S-T extrusions are notable and represent at least a 20 per cent gain over the next highest compressive yield strength.

It is interesting to consider the graph of Fig. 13 in another light. The Alclad 24S-T sheet products shown at the left of the chart illustrate the mechanical properties of products for which most of our current models of military aircraft were designed. In other words, they represent the available material at the beginning of the war. The products of progressively higher strengths towards the right hand portion of the chart illustrate the improvement which has been made available during the war period and gives some prediction of the improved weight characteristics to be expected in newer models.

ACKNOWLEDGMENTS

The author wishes to acknowledge his indebtedness for the development of the data shown in the figures to the technical personnel of the Aluminum Company of America, and particularly to Messrs. W. L. Fink, R. B. Mears, J. A. Nock, Jr. and Miss M. W. Brennecke of the Aluminum Research Laboratories.

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DISCUSSION

Written Discussion: By J. L. Waisman, metallurgist, and W. T. Snyder, assistant metallurgist, Douglas Aircraft Co., Inc., Chicago.

The author is to be complimented on his clear and concise presentation of the newly developed high strength aluminum alloys.

One of the chief disadvantages of the 24S alloy, from the fabricator's point of view, is the fact that its corrosion resistance is sensitive to slight changes in cooling rates. Even with 24S clad material a decrease in the quenching rate not only produces a core more susceptible to intergranular corrosion, but also reduces the difference in solution potential between clad and core. The usual excellent electrolytic protection of the clad for the core is therefore some-

what impaired. From the practical point of view, the above effects necessitate a water immersion quench of non-clad parts and heavier gages of clad sheets. Such quenching produces extremely undesirable distortion in many parts. Is 75S similar to 24S in its sensitivity to slight changes in cooling rate?

Written Discussion: By H. S. Rawdon, chief, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

Papers of the high caliber of this one are rare. Excellence in English and clarity in presentation are among its outstanding features and the paper constitutes a reference of unquestioned reliability on the subject discussed. For fundamental information on the wrought aluminum alloys the structural engineer of aircraft as well as those interested in other applications of these light structural metals need seek no further.

For the reader who is not a specialist in aluminum but who needs to keep abreast of metallurgical developments, Fig. 13, together with the concluding paragraph of the paper, can be highly recommended for obtaining a gist of the paper. These show very clearly and concisely the advances in aluminum structural alloy development from the standpoint of heat treatment and composition. If one also takes into account what the author says (page 147, paragraph 1) concerning some other recently developed alloys (R 301 and Alclad 14S) he has an essentially complete story of the industry.

It is well known, of course, that the heat treatment of the aluminum alloys distinctly differs from the familiar treatments used for steel. The nomenclature evolved for designating the various types of aluminum alloys and their condition with respect to the treatment received has long been a source of admiration for many metallurgists, including the speaker. It is with genuine regret, therefore, that one learns that the advances made in one line of treatment (accelerated aging) have resulted in a change of nomenclature which, at least for some time to come, promises to be quite confusing. The system of nomenclature with which we are familiar was admirably set forth in the article in the Society's Handbook by Messrs. Nagel and Farragher as follows:

"The various tempers are designated by letters following the alloy symbol and separated from it by a dash, for example, A17S-T. Where more than one heat treatment is applied, the different heat treatments may be indicated by a numeral following the letter, for example, 11S-T3.

"The temper resulting from complete heat treatment (including whatever aging is necessary to develop the maximum properties from heat treatment) is designated by the letter 'T'. The letter 'W' is used only with the alloys which require precipitation heat treatment to develop their maximum properties to indicate that they have been subjected only to the solution heat treatment, for example, 53S-W. In some of these alloys there is some aging at room temperatures even though precipitation heat treatment is necessary to develop their higher properties. In these cases, the properties specified for the 'W' temper are those which the material will have after a few days when this aging is practically complete. The soft temper which results from annealing is designated by the letter 'O'.

"The alloys 17S and 24S age spontaneously at room temperatures, consequently there is no 'W' temper"

According to this system of nomenclature the temper characterized by the maximum properties which can be developed by heat treatment is designated by the symbol T. If deformation of the alloy is also necessary as part of the treatment, this can be shown by the use of the additional symbol, R, along with

T. A modification in nomenclature to designate the result of accelerated aging on 24S alloy was evidently necessary since this alloy exists in two important high-strength conditions—(1) normal aging + some cold work and (2) the same material (normal aging + cold work) after accelerated aging. But why choose a designation to indicate the temper of the alloy in the second condition, 80, 81, etc., which bears no resemblance whatsoever to the system with which we are familiar? It would seem that a much less drastic change in nomenclature could have been evolved which would have been more in keeping with the old system and not constitute an additional tax on one's memory.

It will be appreciated if the author will tell us whether the use of symbol T to refer to accelerated-aged alloys in general has been discontinued and superseded by some other symbol or is the new nomenclature relating to 24S alloy the only deviation from the familiar system?

Another change relating to nomenclature, which although it appears in the paper, is not original to it, is the extended meaning of the term "alclad". To the great majority of those who, while not specialists, are familiar with the aluminum alloy system, this term suggests an aluminum-base alloy clad with pure aluminum. However, as set forth in the 1943 edition of the Company's technical bulletin, "Alcoa Aluminum and Its Alloys," the name "alclad" is now used to designate also similar coated products consisting of a base of aluminum-base alloy carrying a coating of an aluminum-base alloy of another kind. Thus, as the author states, Alclad 14S consists of a core 14S alloy (copper, silicon, manganese, magnesium) coated with an alloy of copper-free magnesium-silicide type of alloy.

Possibly the best view to take of this increasing and somewhat inconsistent complexity in nomenclature is that it is part of the price of progress and advance.

But to revert to the paper under discussion, despite these minor adverse criticisms, the speaker considers the paper to be one of which the author, the research laboratory which he represents as well as this Society may well be proud.

Written Discussion: By T. L. Fritzlen, chief research metallurgist, Reynolds Metals Co., Glen Cove, N. Y.

Mr. Dix is to be commended for his excellent summary on "New Developments in High Strength Aluminum Alloy Products", and the extremely interesting historic background of these developments. At the time Mr. Dix prepared his paper, information on the two high strength alloys, R301 and R303, which have been developed by the Reynolds Metals Company, was probably meager or not available, and I would like to take this opportunity of supplementing Mr. Dix's paper with additional information on these two alloys.

R301 alloy is a composite sheet and plate composed of a core alloy having a nominal composition of 4.5 per cent copper, 1.0 per cent silicon, 0.80 per cent manganese and 0.40 per cent magnesium, clad with an alloy consisting of a nominal content of 1.0 per cent magnesium, 0.7 per cent silicon and 0.5 per cent manganese. Hundreds of thousands of pounds of this alloy have been produced for the aircraft industry and has been enthusiastically received by the manufacturers.

The cladding alloy possesses excellent corrosion resistance and provides electrolytic protection to the core alloy, as evidenced by the following potential measurements:

Temper	Core, Mv.	Cladding, Mv.	Difference, Mv.
R301-W	-650 to -660	-830 to -840	170-180
R301-T	-720 to -740	-810 to -830	80- 90

The cladding alloy of R301 responds to solution and aging heat treatments and consequently provides a harder surface and higher strength than that of material clad with pure aluminum.

The percentage of cladding is varied, in order to provide adequate protection against excessive diffusion of the copper into the cladding during prolonged solution heat treatment, from 10 per cent on each face for the gages 0.024 inch and less, to 2.5 per cent on each face on gages 0.102 and heavier.

In addition to the advantage in forming offered by R301-W as mentioned by Mr. Dix, R301-O can be drawn easier than other annealed alloys of similar strength. R301 freshly quenched can be stretcher formed, or drawn easier than other high strength alloys in a similar condition, and R301-T can be formed or dimpled substantially as well as 24S-T alloy.

Another advantage of R301 is the fact that it can be spot welded more easily than other strong alloys, since it has less tendency to crack. With proper technique it is possible to produce spots, which are larger and therefore stronger than those produced in other higher strength aluminum alloys.

R303 alloy is a high strength aluminum alloy containing zinc, magnesium and copper as the major hardening elements and is produced as extrusions, rolled rod, forgings and clad sheet. At the present time, this alloy is being tested by the Government for approval.

R303 alloy is produced in three tempers designated as follows:

- R303-O Annealed.
- R303-T275 Solution heat treated and precipitation heat treated at 275 degrees Fahr. (135 degrees Cent.).
- R303-T315 Solution heat treated and precipitation heat treated at 315 degrees Fahr. (155 degrees Cent.).

Tentative guaranteed mechanical properties for extrusions and sheet are:

Temper	Product	Thickness	Tensile Strength Psi.	Yield Strength Psi. Min.	Elongation Per Cent in 2 Inches Min.
R303-O	extrusions	all gages	35000 Max.	12.0
R303-T275	extrusions	0.040-0.600	75000 Min.	70000	7.0
R303-T315	extrusions	0.040-0.600	70000 Min.	65000	7.0
R303-T275	extrusions	over 0.600	80000 Min.	75000	8.0
R303-T315	extrusions	over 0.600	75000 Min.	70000	8.0
Clad R303-O	clad sheet	all gages	35000 Max.	10.0
Clad R303-T275	clad sheet	all gages	70000 Min.	64000	7.0
Clad R303-T315	clad sheet	all gages	65000 Min.	60000	7.0

It is to be pointed out that clad R303-T275 sheet offers exceptionally higher tensile yield strength. The compressive yield strength has been found equal to the tensile yield strength.

Laboratory corrosion tests, as well as outdoor exposure tests, have shown that clad R303 sheet and R303 extrusions bare, compare favorably to clad 24S-T sheet and 24S-T extrusions. Extensive laboratory tests have shown satisfactory resistance to stress corrosion of clad R303 sheet highly stressed

and severely plastically deformed. The cladding alloy of R303 provides excellent galvanic protection to the core alloy.

The formability of R303 in the "O" temper is equal to that of 24S-O, although slightly higher pressures are required due to the higher yield strength of R303-O.

The formability of R303-T315, which is slightly better than that of R303-T275, approaches the formability of 24S-T alloy and is somewhat better than that of 24S-RT.

Written Discussion: By James W. Poynter, associate metallurgist, Headquarters, Air Technical Service Command, Wright Field, Dayton, Ohio.

Mr. Dix is to be thanked for making available such a complete summary of the present knowledge of the high strength aluminum alloys. The data given should prove to be of value both to the aircraft designer and to the metallurgist.

Aircraft parts made of artificially aged clad 24S alloy described by Mr. Dix in his paper are covered by Army Air Forces Specification No. 11354, Parts, Fabricated; Clad Aluminum Alloy (Elevated-Temperature-Precipitation). The nomenclature used in Revision A of this specification, released October 13, 1943, is the same as that used in this paper, i.e., Clad 24S-T80, Clad 24S-T81, etc. However, Revision B of this specification, released on September 20, 1944, gives a slightly different system of designation.

This system, in common with that used throughout the industry, uses the symbol T to designate the solution treated and aged condition. The numeral 8 is used to indicate cold working followed by an elevated temperature precipitation treatment. A second numeral between the T and the 8 gives the approximate amount of cold working of the solution treated metal prior to the elevated temperature precipitation treatment. The application of this system results in designations such as Clad 24S-T08, for Clad 24S-O material solution treated, followed by an elevated temperature precipitation treatment without intermediate cold work; Clad 24S-T18 for Clad 24S-T material given the elevated temperature precipitation treatment (the numeral 1 representing the approximate amount of cold work resulting from the flattening of the sheet after solution treatment by the producer); and Clad 24S-T68, for Clad 24S-RT material which has been given the elevated temperature precipitation treatment (the numeral 6 representing the approximate amount of cold working given RT sheet).

Specification No. 11354 requires that each part conforming to it be marked in accordance with the above system. This method was selected as standard for the Army Air Forces at the request of the aircraft companies on the basis that it represented a logical sequence of operations which could be expanded if other operations were introduced.

The various tempers of the non-heat treatable aluminum alloys are designated as 2S - $\frac{1}{2}$ H, 2S - etc., where H denotes the final operation (cold rolling) and is preceded by a fraction showing the degree. The same procedure is followed in the designation of 24S-RT where the letter R denotes cold rolling and precedes the symbol T denoting the solution treatment and normal aging.

It is hoped that the producers and users will agree on this system of designation, thus avoiding much confusion.

Author's Reply

I am somewhat overwhelmed by Mr. Rawdon's very kind remarks. I have known him for so many years that I must say I appreciate them.

In regard to the question asked by Mr. Waisman and Mr. Snyder of the Douglas Aircraft Company, the resistance to corrosion of 75S-T is less sensitive to the rate of quenching than in the case of 24S-T. However, there is a sensitivity. In the case of 75S-T the mechanical properties are more affected than the resistance to corrosion so that the ordinary inspection tensile tests will be a check on the rate of quenching and such tests are much easier to perform than are corrosion tests.

In regard to the alclad material, the Alclad 75S-T has an advantage over the Alclad 24S-T in that even with slow quenching the potential between the coating and core is maintained so that the electrolytic protection is present even though the material is slowly quenched.

Mr. Fritzlen's discussion certainly rounds out the picture on the strong aluminum alloy products and I am very glad to have all of this information in one place. I mentioned briefly that the Aluminum Company is prepared to produce Alclad 14S to meet substantially the same specification as R301.

It's going to be of great interest, I am sure, to metallurgists and engineers who follow the shakedown of the various products that are now available and maybe three, or four, or five years from now we are going to know a lot more about metallurgical characteristics and their relation to service performance both as regards resistance to corrosion, fatigue and other properties so necessary in structures.

Both Mr. Rawdon and Mr. Poynter have discussed the nomenclature used in my paper relating to artificially aged 24S. Mr. Rawdon has quoted from the description of the aluminum alloy nomenclature contained in the A.S.M. Handbook.

In building on any system of nomenclature which has already received considerable usage, the problem is how to retain a logical system broad enough for future developments without confusing or changing a well established usage. Consequently, most systems of nomenclature concerning a developing industry are far from ideal and are nearly always a compromise.

The nomenclature system which has been in use by the Aluminum Company of America for some years has suffered to some extent by the conditions just described but has been extended only after very careful consideration of all the complications involved. In its essence it is as described in the A.S.M. Handbook and I would call particular attention to the sentence "where more than one heat treatment is applied the different heat treatments may be indicated by a numeral following the letter, for example 11S-T3". T3 indicates a solution heat treatment plus working. In the same manner, T8 indicates a solution heat treatment followed by working, followed by elevated temperature precipitation.

When 24S was first developed the use of artificially aged tempers was not contemplated and therefore, again in agreement with the A.S.M. description, the temper resulting from complete heat treatment was designated by the letter T. When the use of the artificially aged tempers was recently accepted by

the industry there was a tremendous amount of data and information on 24S-T. Consequently it was impossible to use the T alone to indicate the artificially aged temper. Further, the mechanical properties obtained after artificial aging varied with the amount of cold work which the product had received subsequent to solution heat treatment but prior to artificial aging. Thus, the artificially aged conditions fall in the T8 temper classification previously described. The additional numerals 0, 1, 4, and 6, indicating approximately the amount of cold working, seemed to be a logical and simple addition.

Mr. Rawdon has suggested the use of the symbol R along with T to indicate the cold working. However, R in the past has indicated a specific degree of cold working such as is given to 24S-RT sheet. Any use of R and T would confuse the issue in regard to all of the previous data on the 24S-RT product which of course is not artificially aged.

Mr. Poynter's information, that the Army Air Forces have changed the nomenclature accepted for Revision A of Army Air Forces Specification 11354 when they issued Revision B, is quite disturbing. This precipitate change violates the first principle of establishing a satisfactory nomenclature system, which is that once a system is put into operation it should not be changed without very careful consideration of the effect on the scheme as a whole. The change conflicts in other respects with the well established scheme of nomenclature of one of the principal producers of the products. It is unreasonable to expect a producer to permit his whole scheme of nomenclature to be upset by any group that desires to change one part of it without considering the effect on the whole. On the other hand, undoubtedly the producers would be glad to co-operate with a responsible authority to consider any necessary changes in aluminum nomenclature.

Mr. Rawdon has commented on the use of "alclad" to cover the products other than those having a pure aluminum coating. The trademark "Alclad" covered duplex aluminum articles and was not confined to any particular combination. Although the Aluminum Company has given up claim to the trademark, the word is used to describe clad products in which one of the principal functions of the coating is to improve the resistance to corrosion of the product. For some time Alclad (72S)3S sheet, in which the coating is an alloy which will electrolytically protect the core, has been used for products requiring resistance to perforation, such as aircraft gasoline tanks. The alclad coating on Alclad 75S is also an alloy coating, hence the selection of "Alclad 14S" to describe a 14S core clad with a corrosion resistant alloy of copper-free magnesium silicide type seems to be a logical selection.

It will be evident to readers of these several discussions that the aircraft engineer has two new types of aluminum alloy products available for use in high strength, light weight structures, the duralumin-type alloys R301 and Alclad 14S-T, and the new aluminum-zinc-magnesium type alloys 75S and R303. The latter alloys represent the highest strength aluminum products commercially available and the former, although not as strong, possess better shop characteristics.

Our appraisal of the relative formability of these two types of alloys differs somewhat from that given by Mr. Fritzlen. The relative formability of the products under consideration is well illustrated in the following tabulation:

Minimum Bend Radii for Heat Treated Alclad Sheet
90-Degree Power Bends for Fourteen-Gage Sheet (0.064 Inch)

Alloy	Temper	Minimum Bend Radii
Alclad 14S-W	As-quenched and refrigerated	1T
Alclad 75S-W	As-quenched and refrigerated	1T
Alclad 24S-W	As-quenched and refrigerated	1½T
	Aged at Room Temperature	
Alclad 14S-W	114 Days	1½T
Alclad 75S-W	14 Days	2T
Alclad 24S-T	14 Days	2T
	Aged at Elevated Temperature	
Alclad 14S-T	10 hours at 340 degrees Fahr.	3T
Alclad 75S-T	24 hours at 250 degrees Fahr.	4T

While it is recognized that bend radii are not by any means the whole story on forming, they are at least a measure of one type of forming and our other shop experience would rate these alloys and tempers in about the same order as the bend radii in the tabulation. Thus, immediately after quenching we would rate Alclad 14S-W and Alclad 75S-W as being comparable in forming characteristics and somewhat better than Alclad 24S-W. Alclad 14S-W after room temperature aging we would rate superior to Alclad 24S-T. Alclad 75S-W after about 14 days of room temperature aging we would consider somewhat comparable with Alclad 24S-T. In the fully aged temper we would rate Alclad 14S-T as definitely inferior to Alclad 24S-T and Alclad 75S-T inferior to Alclad 14S-T.

In regard to Mr. Fritzlen's comments on spot welding, the work in our welding laboratory on Alclad 14S-T does not show it to have less tendency to crack nor to produce higher strength spots than Alclad 24S-T. It is questionable if such broad generalizations can be made without going into greater detail than is permissible here.

Only time and experience will prove the value of the different strong aluminum alloy products and establish the applications in which each will prove the most economical. The author, for one, expects to observe with much interest their use in the war and postwar periods.

ALUMINUM ALLOY FORGING MATERIALS AND DESIGN

By L. W. DAVIS

Abstract

The rapid expansion of the aluminum forging industry in recent years has been accompanied by improvements in materials available for forgings and by development of more accurate knowledge regarding proper aluminum forging design. Very little information on this subject has appeared in technical literature.

The alloys available for forgings are enumerated and the advantages of each are indicated. A method of determining relative forgeability is described. This method permits evaluation of new forging materials and accurate comparison with other alloys.

New forging equipment has been developed especially to handle aluminum alloys. A description of a hydraulic forging press, a forging roll and other equipment is given.

The necessity for controlling flow lines and methods for accomplishing this control by means of forging technique and die design are shown. General rules for the design of aluminum forgings are enumerated, including draft angles, location of forging plane and parting line, size of fillets and radii and allowance for shrinkage. The effect of proper and improper design is demonstrated on typical forgings.

ALUMINUM ALLOY FORGING MATERIALS AND DESIGN

FOR several years the use of aluminum forgings, particularly in aircraft, has been undergoing a very rapid expansion. In fact the productive capacity of the country today is at least 70 times the capacity existing 5 years ago. Probably about 12 to 13 per cent of the metal produced in the country today eventually reaches the trade in the form of forgings. Depending upon the size and type of airplane, aluminum forgings represent from 10 to 18 per cent of the finished aluminum weight present in our military aircraft. This tremendous expansion has speeded up the normal process of evolution both in the development of forging materials and in the development

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of equipment for the production of forgings. Little information regarding these developments has found its way into technical literature and, therefore, these facilities have not always been employed as advantageously as possible.

In discussing aluminum alloy forgings, no attempt will be made to go into detail concerning production methods except to point out some of the controls that are essential if a quality product is to be obtained. With the development of the art, the intricacy of the parts produced has increased materially. Although many things have been, and still can be, accomplished in this respect, there are certain limitations which should be appreciated. Some of these limitations as well as the possibilities of this type of product will be discussed.

STANDARD FORGING ALLOYS

In Table I, the commercial wrought alloys of aluminum that are used today in production forgings are shown with their nominal chemical composition. It will be noted that of these 9 alloys, the first 5 contain 4 to 5.5 per cent copper placing them in the group familiar to all. One alloy, 32S, contains about 12.5 per cent silicon with small amounts of other alloying elements. The next 2 alloys are in the aluminum-magnesium-silicide family, and the last alloy shown, 70S, is one of the aluminum-zinc group with small amounts of other alloying elements.

Reference to Table II will indicate the mechanical properties obtained from these alloys. In the aluminum-copper series of alloys, 14S-T possesses the highest ultimate strength and also develops a considerably greater Brinell hardness than any of the other materials. The use of this alloy has expanded more rapidly during the war than any of the other older materials because of the high mechanical properties which have made it a very suitable alloy for aircraft structural purposes. The use of 17S-T has not increased at the same pace as the other alloys for several reasons. Where high strengths are desired, 14S-T is used instead of 17S-T. It will also be noted that 25S-T has the same mechanical properties as 17S-T. This alloy is more easily worked at forging temperatures and it does not age at room temperature as does 17S-T, thereby making it a more desirable forging alloy. The advantages left with 17S-T are better machinability and somewhat better resistance to corrosion. Recent tests have indicated that 14S-W, with mechanical properties the same as 17S-T,

Table I
Nominal Composition of Aluminum Forging Alloys

Alloy	Percentage of Alloying Elements				Aluminum and Normal Impurities—Constitute Remainder				
	Copper	Silicon	Manganese	Magnesium	Zinc	Nickel	Chrom.	Lead	Bismuth
11S	5.5	0.5	0.5
14S	4.4	0.8	0.8	0.4
17S	4.0	...	0.5	0.5
18S	4.0	0.5	...	2.0
25S	4.5	0.8	0.8
32S	0.9	12.5	...	1.0	...	0.9
A51S	...	1.0	...	0.6	0.25
53S	...	0.7	...	1.3	0.25
70S	1.0	...	0.7	0.4	10.0

Heat treatment symbols have been omitted since composition does not vary for different heat treatment practices.

Table II
Mechanical Properties of Aluminum Alloy Forgings

Alloy	Minimum Specification Values			Typical Values (Not Guaranteed)			
	Yield Strength (Set=0.2%) Lb./sq. in.	Tension— Ultimate Strength	Elonga- tion Percent in 2 Inches	Hardness Brinell 500 Kg. Load 10 mm. Ball	Shear Strength Lb./sq. in.	Fatigue Endurance Limit Lb./sq. in.	Density Lb. per cu. in.
11S-T	34,000	55,000	12.0	90	31,000	13,000	0.101
14S-W	30,000	55,000	16.0	100	36,000	15,000	0.101
14S-T	55,000	65,000	10.0	130	45,000	16,000	0.101
17S-T	30,000	55,000	16.0	100	36,000	15,000	0.101
18S-T	35,000	55,000	10.0	100	38,000	14,500	0.103
25S-T	30,000	55,000	16.0	100	35,000	15,000	0.101
32S-T	40,000	52,000	5.0	115	38,000	14,000	0.097
A51S-T	34,000	44,000	14.0	90	32,000	10,500	0.097
53S-T	30,000	36,000	16.0	75	24,000	11,000	0.097
70S-T	40,000	50,000	16.0	85	37,000	19,000	0.107

is at least equivalent to 17S-T in resistance to corrosion. Therefore, it is possible that in the near future 14S-W will be permitted as an alternate material. This means that 17S-T will disappear from the active list of forging materials. 11S, which is the alloy that contains the lead and bismuth additions, has had very limited forging application, since it is used only for those applications where its superior machinability makes it the most desirable material.

As mentioned above, 25S-T is desirable because it does not age harden at room temperature. This means that forgings can be straightened in the quenched condition (heat treated, but not aged) with little difficulty. This is the alloy employed today for most propeller blade forgings as well as for many smaller applications. It is one of the oldest forging alloys, but is still used for a very large proportion of the total forging tonnage.

18S-T and 32S-T are used for forged parts in which the retention of strength at elevated temperature is essential. Pistons and air-cooled cylinder heads for aircraft engines are the principal products for which these alloys are employed although there are other applications. Alloy 32S also has the advantage of a lower coefficient of thermal expansion than any other wrought aluminum alloy.

Where low cost is a primary consideration, alloys A51S and 70S are usually used. Both of these alloys can be forged much more readily than 25S, making for lower production cost. Because of this excellent forgeability, large and intricate parts such as radial aircraft engine crankcases are made from A51S-T while they would be difficult and much more costly to produce in some of the harder alloys.

In order to determine the relative forgeability of these and other alloys at various elevated temperatures, a test was devised for a comparison of different materials. The method consists in a measurement of the resistance of the various alloys to impact compression without lateral restraint. A comparison of the decrease in height of specimens of one alloy at different temperatures, or of different alloys under similar conditions, yields a relative classification of the capacity of the various alloys to submit to forging operations such as upsetting and direct compression where the material is not appreciably constrained nor is it forced to extend axially.

There are probably many metals which have approximately equal resistance to impact compression or tension or at least values of the same order of magnitude. Where this is the case, the drop hammer method results may then be considered representative of the general forgeability of that material or its alloys. Iron and copper can be easily drawn, upset and compressed. However, magnesium and zinc are compressed readily and yet extend with considerable difficulty during forging, making this type of test of doubtful value in the case of these metals. Commercial practice has proven that the aluminum alloys can be both easily depressed and drawn by the drop hammer at elevated temperatures.

The drop hammer test was selected for the investigation of the plasticity of wrought aluminum alloys at elevated temperatures because of its simplicity and the ease with which it can be reproduced in the forging plant without the addition of any special equipment. The test uses cylindrical slugs of uniform diameter and height which can be upset in a board hammer employing a known weight, travelling

a known distance, and thereby developing a blow energy of a definite number of foot-pounds. From this test a set of values can be derived indicating the change in dimensions compared with forging temperature, and it is possible to calculate the resistance to deformation in pounds per square inch at any temperature. The results of tests reveal why intricate forgings can be produced in A51S alloy. For a proper interpretation of the values obtained, the temperature at which the various alloys become hot short must be known since this point determines in practice the maximum forging temperature. The hot-short temperature varies for the different aluminum alloys depending on the chemical composition. Alloys A51S, 70S, 25S and 32S can be forged at temperatures up to about 875 degrees Fahr. (470 degrees Cent.). For 14S, 17S, and 18S, the optimum temperature is a little above 800 degrees Fahr. (425 degrees Cent.). Then going to the harder materials such as Y alloy and 24S the temperature is reduced to about 750 degrees Fahr. (400 degrees Cent.). A set of values from the drop hammer test is helpful in the evaluation of new forging materials since it is a fairly simple matter to determine a few points and see how they compare with the figures for existing alloys, and thus decide whether there is any possibility of using that composition in commercial forging work. It is also possible to make a reasonably accurate estimate of the type of forging equipment that must be used if a new composition possesses properties that give it definite advantages over other materials for certain applications. Proceeding from the alloys of relatively high forgeability to the materials more difficult to work, the value of the forging hammer decreases, the size of hammer that must be used for a given forging increases, and the importance of mechanical and hydraulic presses in place of hammers also increases.

This is one of the reasons why pistons are made in horizontal forging machines or presses rather than in hammers, and also why cylinder heads are forged in mechanical presses rather than in hammers. These tests also point to the fact that exploitation of difficult forging materials such as Y alloy requires the use of very large press equipment.

FORGING EQUIPMENT

It will be appreciated from the above why it has been necessary to parallel the development of forging alloys with the de-

velopment of types and sizes of forging machinery and accessory equipment capable of making the products. In some cases, this has meant simply increasing the size of existing types of equipment and in other instances entirely new kinds of forging machinery have been developed.

When work on the new aluminum-zinc-magnesium alloys was first undertaken, it was found that several of the most desirable alloys in this group could not be rolled from the type of ingot generally used for making rolled products. This meant that the ingots had to be broken down by some type of forging operation. It was soon discovered that hammers could not be used for this purpose since the cast structure of these alloys would not withstand an impact blow without cracking. Preliminary experiments indicated that the squeezing action of a hydraulic press was the type of working that would be successful with these alloys. There were no presses in the aluminum forging industry at that time, but it was possible to obtain the use of the conventional type of steam-hydraulic forging presses in some of the plants in the steel forging industry.

At about the same time the forged aluminum alloy piston for Diesel engines was being developed. This piston was too large to make on any horizontal forging machine or upsetter available at the time, and its design was such that a hammer was out of the question. Consequently, this forging also was made on a steam-hydraulic forging press in a steel forging plant.

It soon became evident that the type of forging presses then in use in the steel industry were not particularly well suited to the requirements of aluminum. Aluminum should be worked with a comparatively rapid pressing speed and long power strokes can be used. The steam-hydraulic presses had a sufficiently rapid travel, but in most cases were limited to a 4 to 5-inch power stroke. A full hydraulic press could deliver the long working stroke desired, but in the past these presses had been built for comparatively low pressing speeds. Also it was found that the load should be concentrated on a smaller, stiffer platen for aluminum than had been the custom in steel forging press construction. If the press is to be used for closed die work, the alignment must be much better than was necessary on most existing presses. As a result, an entirely new type of hydraulic forging machine was designed and built. This was a full hydraulic press capable of a speed as high

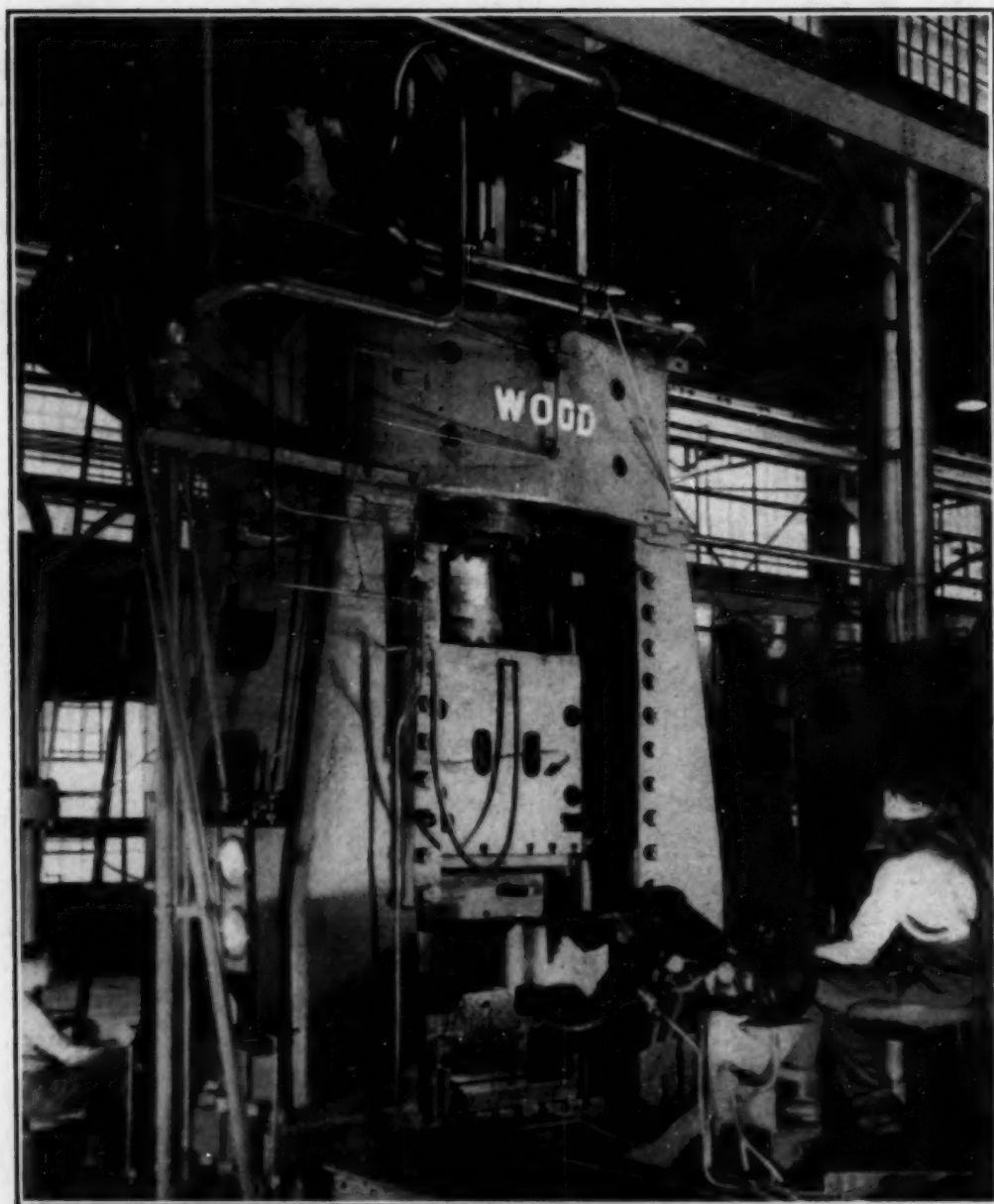


Fig. 1—3000-Ton Hydraulic Forging Press and Manipulator Engaged in Cogging an Aluminum Ingot.

as 7 inches per second under full load, with a power stroke of as much as 36 inches if necessary. It has a small platen, very accurate guiding, and a valve control system entirely new to the forging industry, imparting unusual accuracy to the length of the power stroke. Fig. 1 shows one of these presses drawing down on aluminum ingot.

The manipulators used with the steel forging presses were built to handle heavy ingots or billets and were not as flexible or

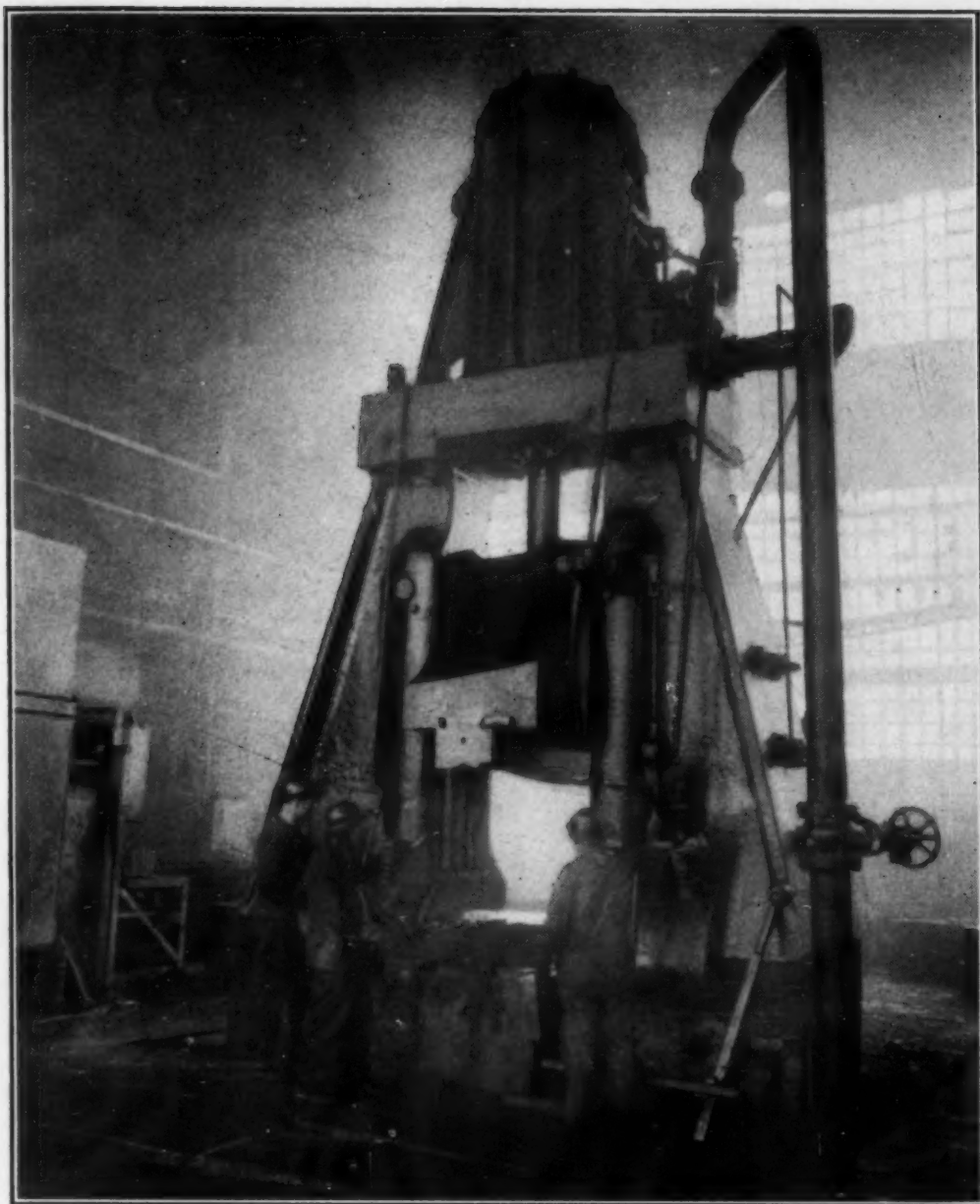


Fig. 2—35,000-Pound Steam Drop Forging Hammer Working on an Aluminum Propeller Blade.

as fast as was desirable for aluminum. Accordingly, the manipulators serving the aluminum forging presses were of a new design with features especially adapted to handling light metals. A portion of the manipulator can be seen in Fig. 1 in front of the press holding the ingot as it is forged.

With the demand for larger and more complicated forgings in alloys that were more difficult to forge, it became obvious that larger forging hammers were essential. In the aluminum industry

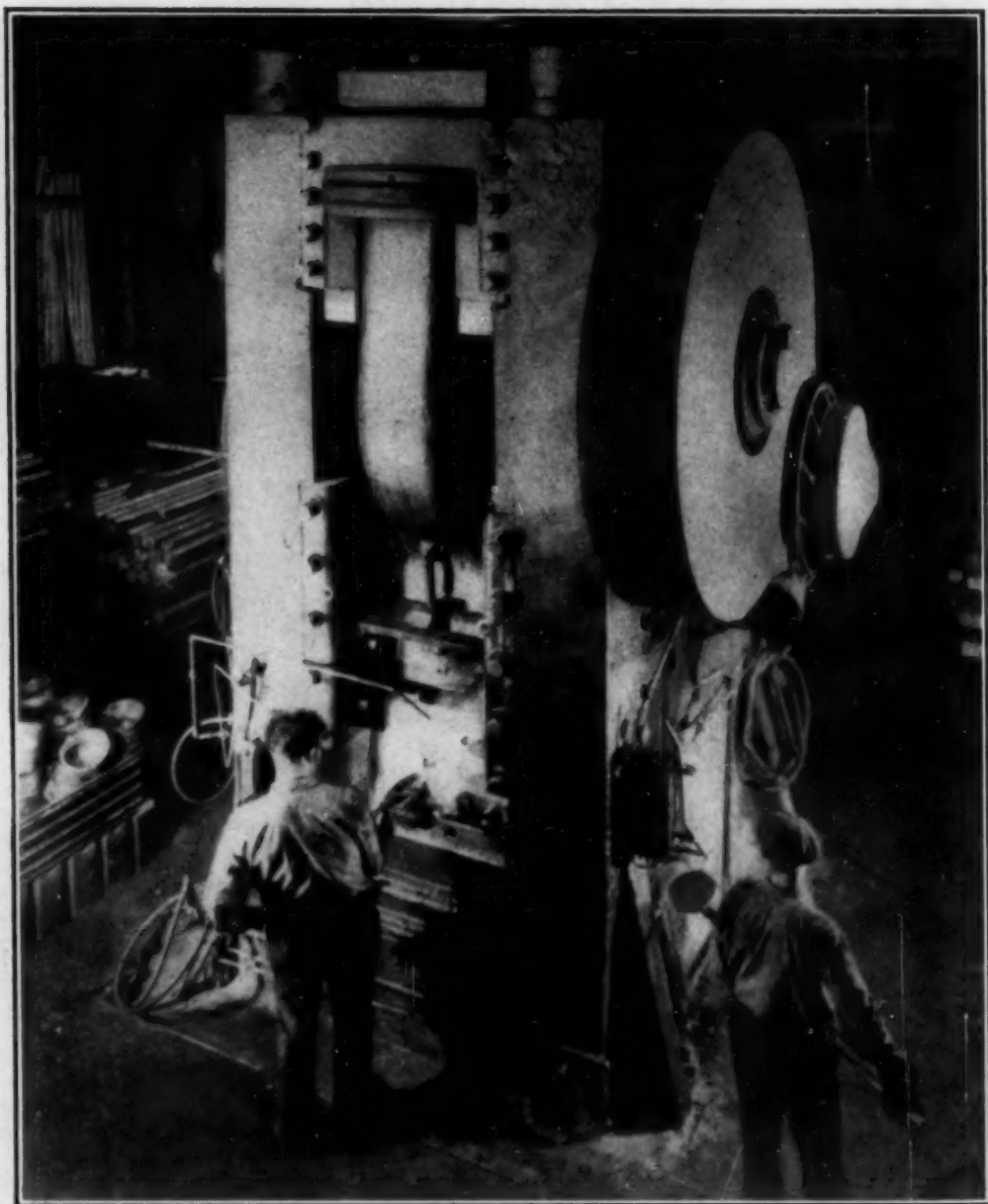


Fig. 3—Mechanical Forging Press About 1500-Ton Capacity Working on Aluminum.

and also in the steel industry, hammers of 18,000 to 20,000-pound capacity were the largest pieces of equipment available in most shops. In fact, few hammers of larger size had been built. A hammer of 35,000-pound capacity was designed, and one of the first hammers of this size to be built was installed in an aluminum forge shop. A hammer of this type is shown in Fig. 2. A number of these hammers are in operation in aluminum forge shops throughout this country today, as well as many hammers of

25,000-pound capacity. Without these it would have been impossible to produce many of the large forgings in use today, including propeller blades for big bombers, crankcases for the largest radial aircraft engines and numerous airframe fittings.

Upsetters and mechanical forging presses are not new types of equipment since both have been used by the ferrous and non-ferrous forging industries for years. The interesting fact is that



Fig. 4—8-Inch Upsetter Used for Making Forged Aluminum Air-Cooled Cylinder Heads.

the demands of the aluminum forging plants have brought about the development of larger and stiffer machines of both types. The 2500-ton forging presses that were built especially for the production of forged cylinder heads have a longer stroke, a larger fly-wheel and other refinements not found on the standard 2500-ton press prior to that time. Some plants are using presses of 4000-ton capacity for work on cylinder heads and other heavy forgings. Fig. 3 shows a somewhat smaller press (about 1500-ton) working on aluminum forgings. In Fig. 4 is seen one of the large upsetters used in producing cylinder heads. Machines of 8 and 9-inch capacity are required for this work, and many of these machines are available and are being used in many aluminum forging plants today.

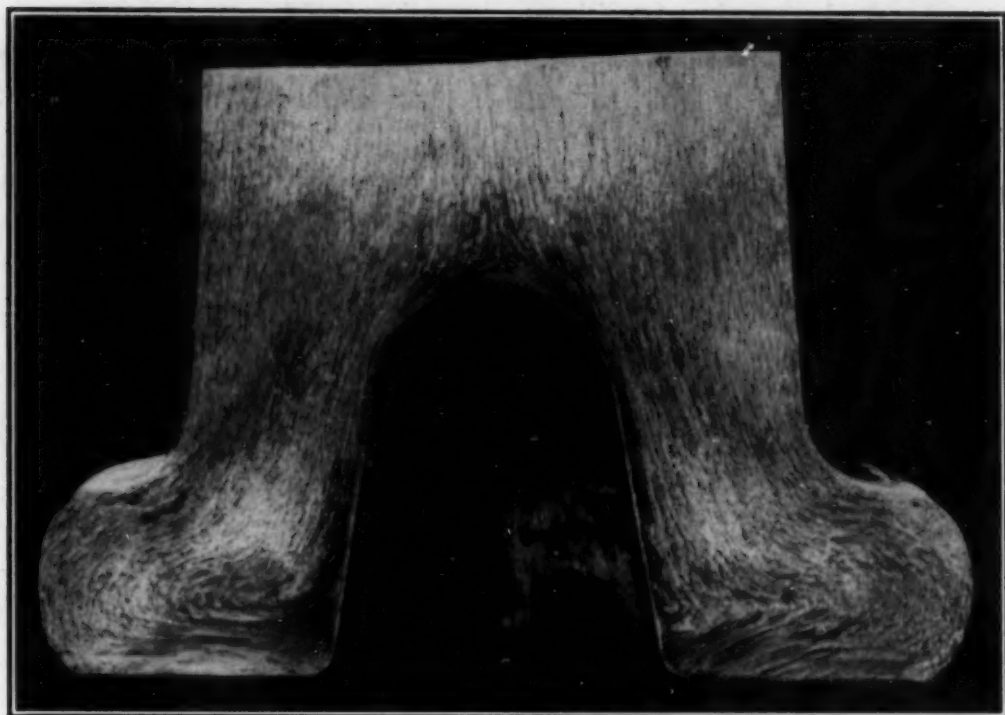


Fig. 5—Macro-Etched Cross Section of the Hub Portion of a Variable Pitch Propeller Forging. 25S-T Alloy.

ALUMINUM FORGING DESIGN

In order that dies may produce the number of pieces that they should, the most careful attention must be paid to die design and correspondingly to forging design. In any discussion of design, one of the first points to be considered is the establishment of the proper grain flow lines in the part. All forgings are possessed of a characteristic structure imparted to them by operations incident to the production of the original forging stock such as rolling, extrusion or casting, as well as by the subsequent forging operations themselves. By the proper selection of stock and sequence of operations, this structure can be controlled and developed to conform to the shape and functioning of the part in question.

Proper die design is a necessity in obtaining satisfactory grain flow in forgings. Many forging imperfections are the result of improper grain flow in the section in question. Frequently, simple changes in die design or forging technique are sufficient to correct the flow and eliminate the difficulty. It is important, therefore, that in parts subjected to high tensile, torsional and repeated impact stresses, the grain flow follow the proper direction so as to

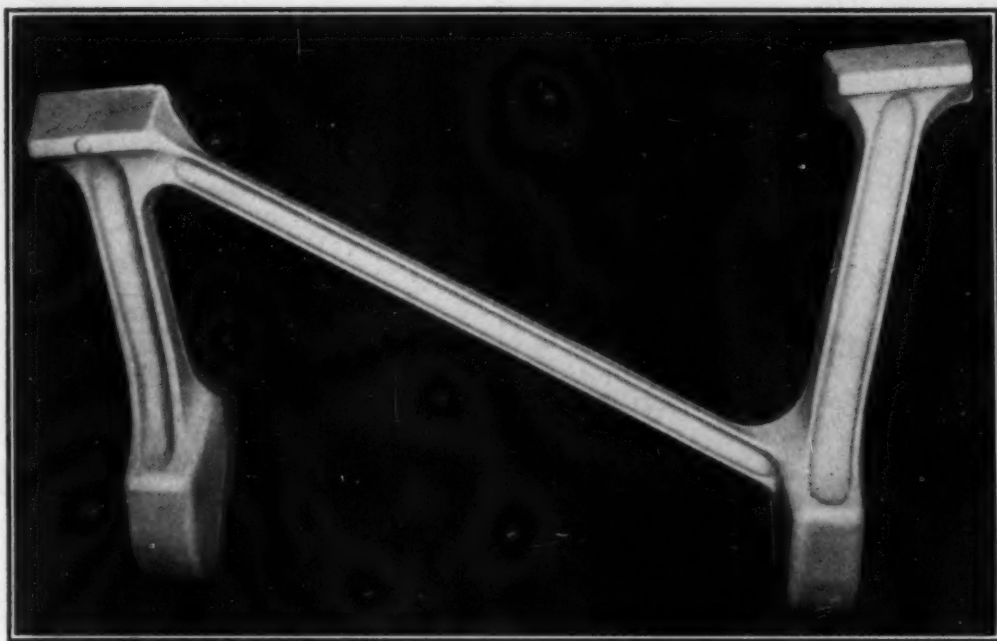


Fig. 6—Large Airframe Structural Fitting of Unusual Design. 14S-T Alloy.

afford the maximum strength at the point of maximum loading.

The establishment of proper flow lines in forgings can be explained best by considering a few concrete examples. Fig. 5 is a macroetched cross section of the hub portion of a variable pitch propeller forging. It will be noted that the grain flow follows the forging line smoothly and without kinks. It is also important in this case that the flow lines should remain the same to such a depth that they will also conform to the finish machined contour of the propeller. Excess metal is permitted to flow freely in a lateral direction increasing the diameter of the base. This is removed during machining without disturbing the grain contour of the finished part.

Fig. 6 is a very unusual looking airframe structural fitting. It is shaped like a letter "Z" with a heavy section at each corner and very light connecting legs. It is obvious that this part could be produced with simple dies from a piece of plate. This would result in flow lines that would follow either the two outer legs or the center leg and cut through the other parts of the forging at an angle. Since the user specified flow lines following all three legs because of the service loading of the part, it was necessary to develop a die design that would accomplish this result. A blank was prepared and placed in the die shown in Fig. 7, and the pre-

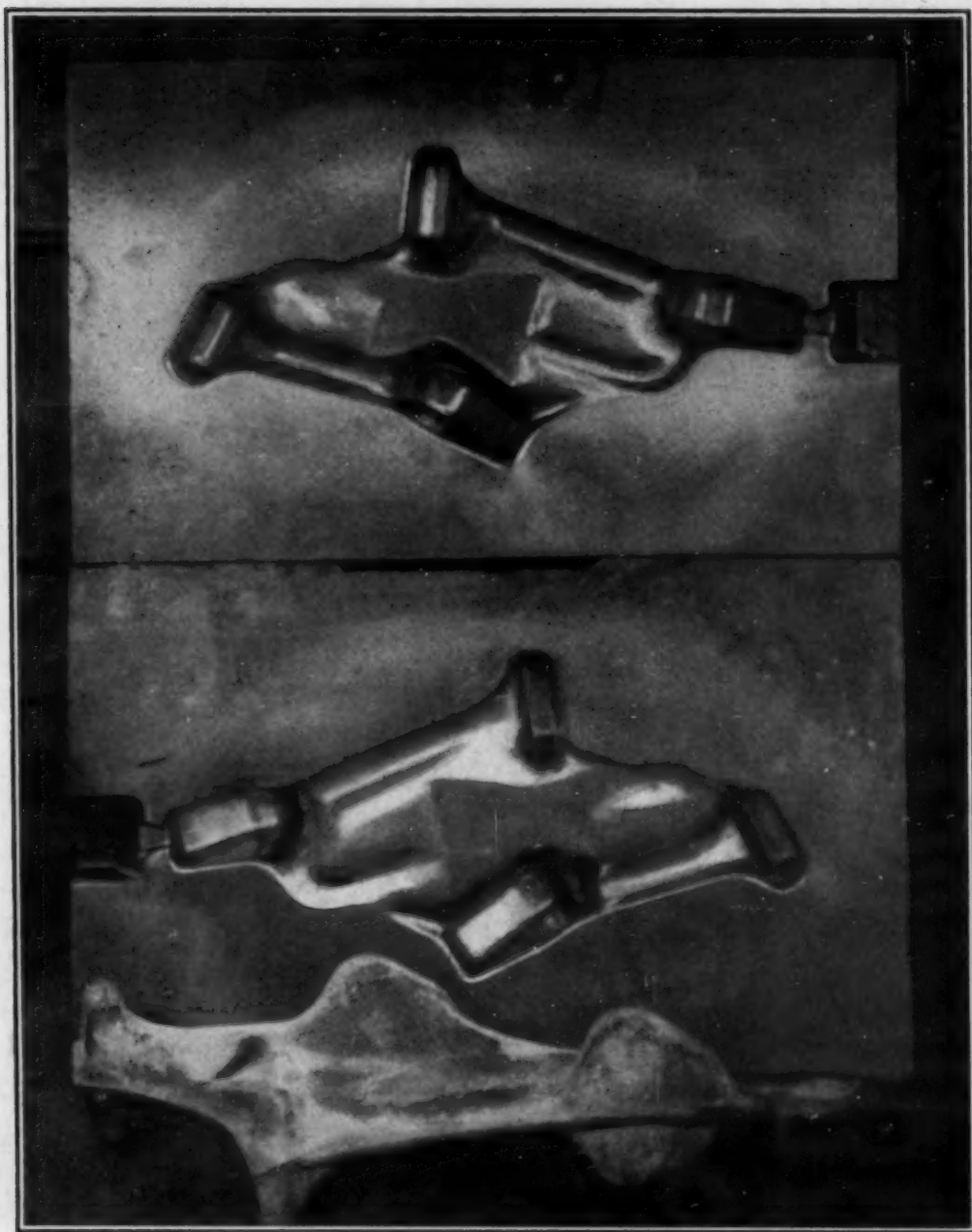


Fig. 7—Preliminary Blocking Dies for Part Shown in Fig. 6.

liminary blocked piece was then forged in the blocking die shown in Fig. 8. It will be seen that this produces a piece with the metal for the three legs parallel to each other and in line with the flow lines of the bar stock that was used. This piece was opened out by bending the junction of the center leg with the two corners so that it was approximately the shape of the finished part and could be placed in the finishing die (Fig. 9). This resulted in the de-

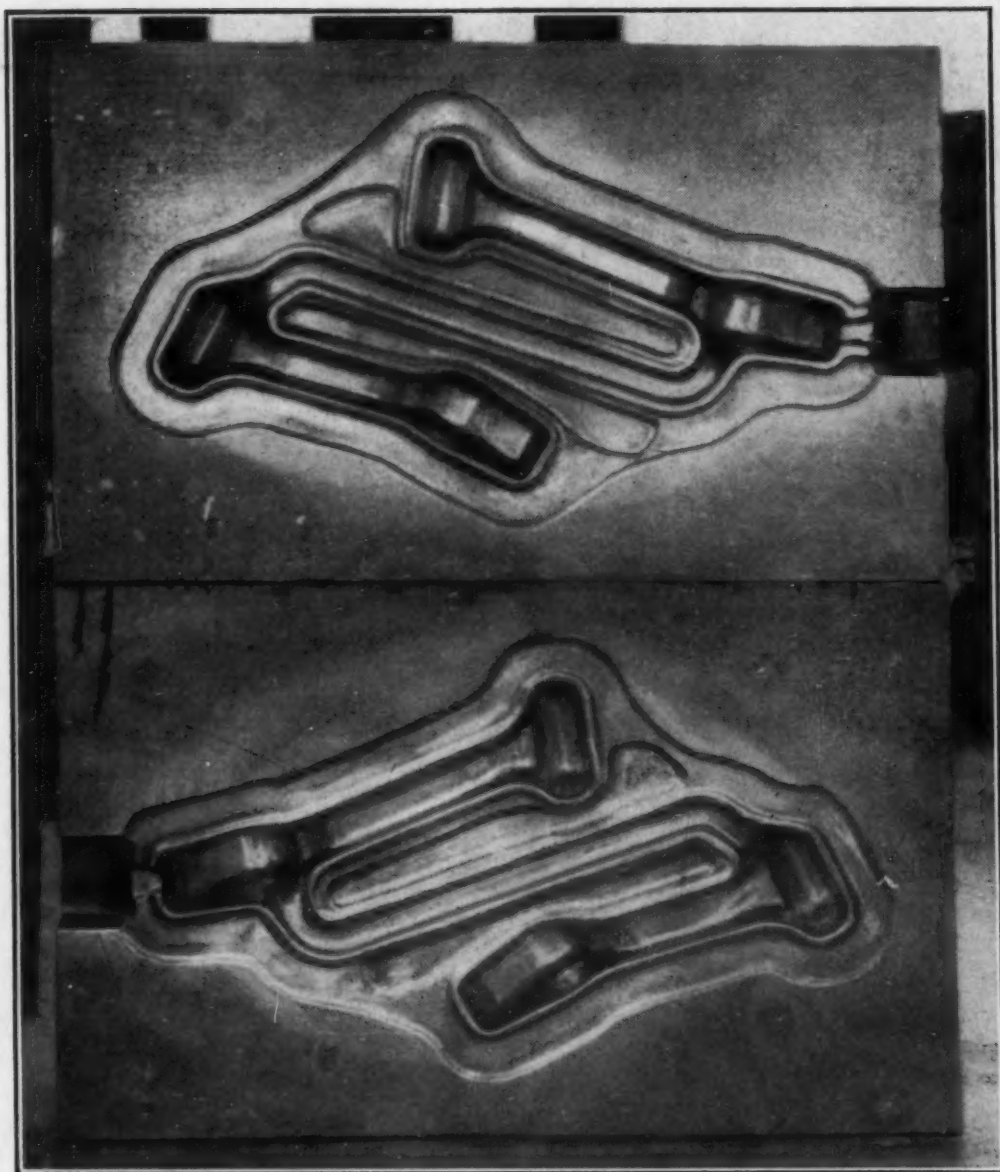


Fig. 8—Blocking Dies for Part Shown in Fig. 6.

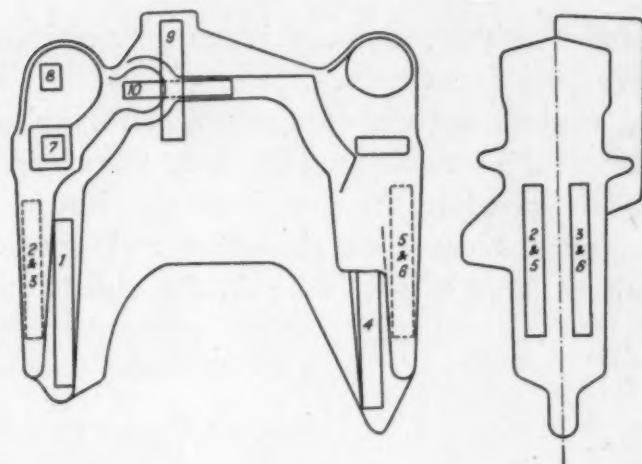
sired flow throughout the entire forging. With a knowledge of the service conditions, a study of this kind should be made before any forging is produced so that the proper sequence of operations can be incorporated in the dies.

Regardless of the care exercised in die design and in the use of the most satisfactory type of stock, variations in tensile properties will still exist in most forgings when tests are taken in different directions. Fig. 10 shows the outline of a large airframe fitting weighing about 55 pounds and produced in 14S-T alloy.



Fig. 9—Finishing Dies for Part Shown in Fig. 6.

The locations of 10 tensile test specimens are indicated on the outline and the properties obtained from these specimens are shown in the table below the sketch. In this forging it was possible to obtain transverse bars in two directions at right angles to each other as well as to the longitudinal bars. It will be noted that the tensile strength and yield strength are about the same in all directions. However, there are large variations in the elongation. This is particularly noticeable in bars 7 and 8 which are perpendicular



Test Bar No.	Tensile Properties			
	T. S. Psi.	Y. S. Psi.	Per Cent Elongation	B.H.N.
1	68,100	60,200	11.5	136
2	70,000	62,000	11.0	130
3	70,600	62,700	12.0	136
4	69,600	61,500	12.0	143
5	70,600	62,400	10.0	136
6	67,900	60,900	8.5	130
7	65,900	61,700	2.5	143
8	65,000	60,800	2.5	143
9	68,600	61,300	6.5	143
10	68,300	61,000	9.0	143

Fig. 10—Outline of 55-Pound Structural Fitting (14S-T Alloy) Showing Location of Test Bars. Tabulation Gives Tensile Properties of Test Bars from Indicated Locations.

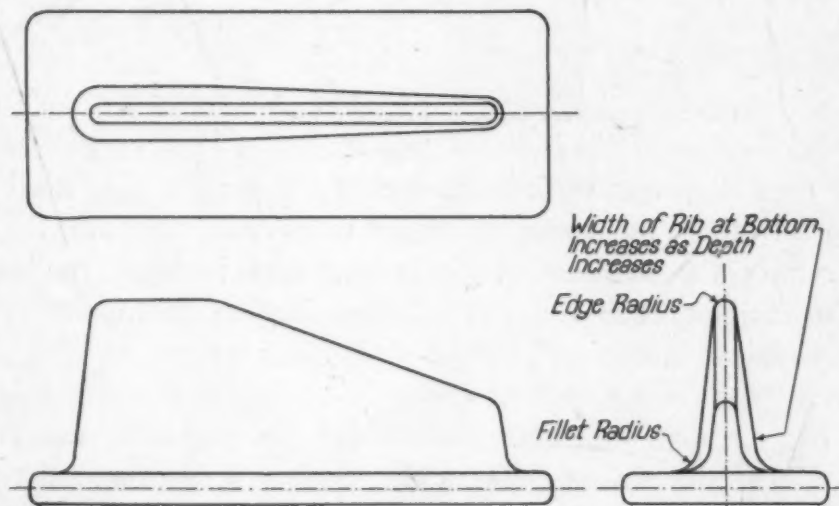


Fig. 11—Method of Designing a Rib of Varying Height so that the Die Impression Can Be Produced with a Single Cutter.

to the parting line of the forging and in a very heavy section. Here the elongation drops off to 25 per cent of that specified for the alloy on a longitudinal test bar. This condition exists in many forgings and is not at all alarming since it does not affect the

serviceability of the part, the maximum properties having been provided where they are required.

With the general outline of the finished forging determined and the sequence of operations established, there are certain rules that should be followed in the design of the forged part. Draft angles, fillets, edge radii, web thickness and general tolerances should be made as large as possible. At the same time the forging

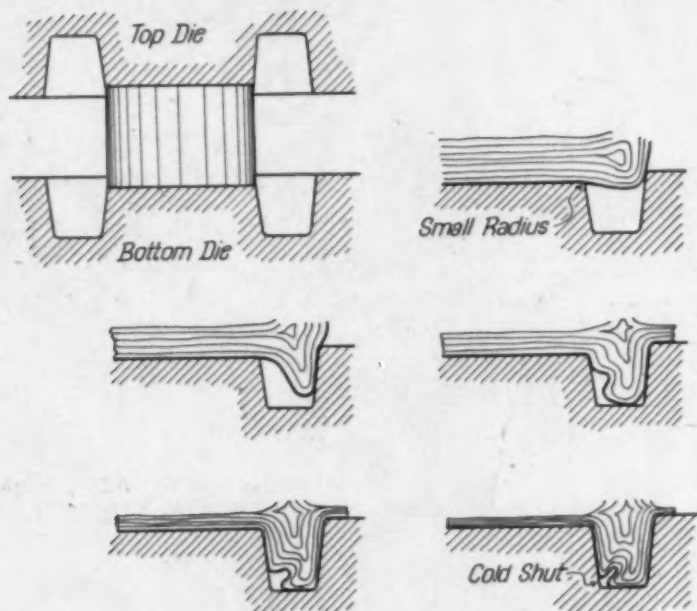


Fig. 12—Demonstration of Formation of a Cold Shut Due to Insufficient Radii in Forging Die.

should be well proportioned and a straight parting line used whenever practical. Adherence to these rules will improve the metal flow, reduce the number of operations and increase the die life.

To facilitate the removal of the forging from the finishing die impression, a slight taper or draft is required on the side of the cavity for most types of forging. When indicating this draft on a forged part, consideration should be given to the requirements of the die sinker. Every angle on the forging must be reproduced in the steel die and, therefore, changing cutters too often for various draft angles or radii should be avoided. This is illustrated by the simple type of forging shown in Fig. 11. Here is a rib of varying height that will be finish-machined to uniform thickness. The draft angle, fillet radius and edge radius are kept constant along the entire rib so that it can be sunk in the die without changing the cutter. This results in a rib of varying thickness

but no machining difficulties are introduced and die cost is materially decreased.

When ribs or bosses are opposite each other in the two halves of the die and they are not of the same height, the deeper impression is then increased so that the two sides match at the parting line. This is desirable so that a good trimming line can be obtained on the forging.

Many forgings can be placed in the die so that the shape of the part produces natural draft and no additional finish is added.

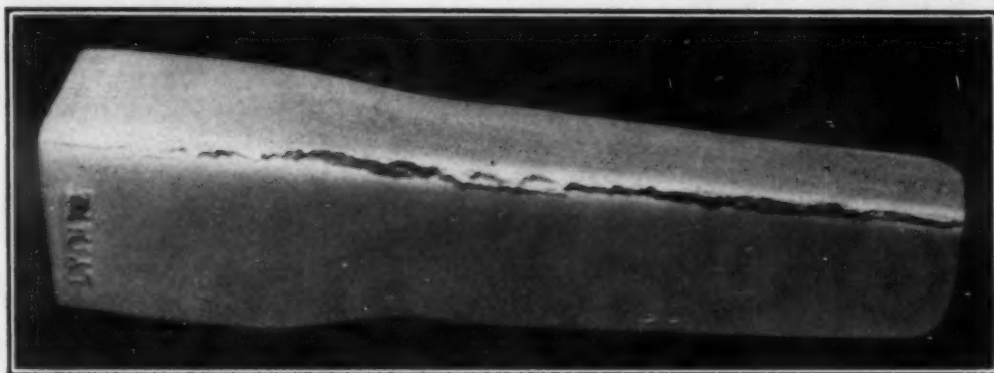


Fig. 13—Forging with Sharp External Radius.

This should be done whenever it is consistent with good forging practice, since machining on the finished part can be eliminated in this way.

The number of forging operations necessary to convert stock to the final shape of the forging depends entirely on the design of the forged part. One of the most important rules to follow in designing forgings is that even though abrupt changes in section cannot be avoided, generous radii and fillets should always be provided. Large radii assist in the flow of aluminum in its plastic state and prevent the formation of laps, folds and other defects in the finished forging.

Statements have been made that small folds occurring in steel at high forging temperatures may weld together and disappear. In the case of aluminum alloys, there is always a transparent oxide film on the surface of the metal and, therefore, no welding of the surfaces within a fold is possible. Also there is no scaling of the surface, and the elimination of small surface defects into the scale is impossible.

A natural question for the designer to ask is, "What harm

can be done to a forging by the use of small radii if they are desirable in the finished part?" The series of sketches in Fig. 12 demonstrates the formation of a fold or cold shut in a projection on a forging because of insufficient radii. Because of the sharp corner, the metal does not flow along the inside of the projection, but shoots across to the outer wall and then pivots around and flows back on itself creating a defect.

Sharp radii on the outside of the forging, and particularly in the bottom die, have quite a different effect, but one which is equally undesirable. Fig. 13 shows a forging that has caused a

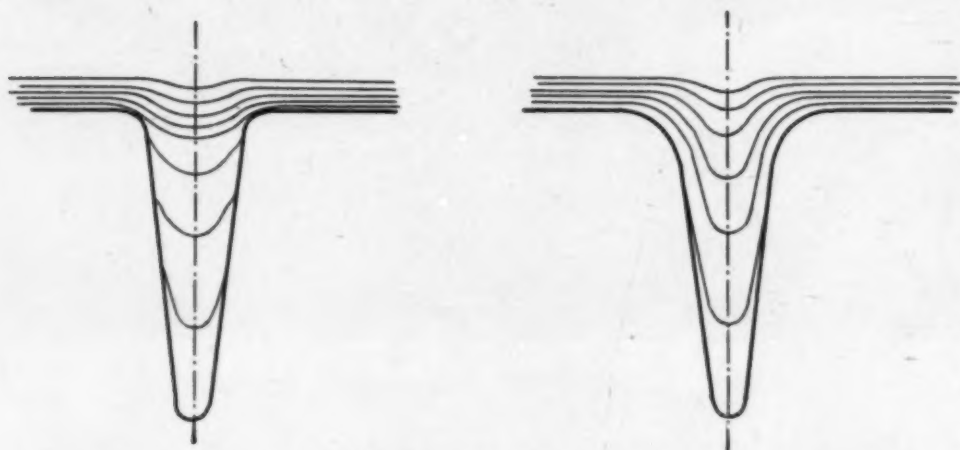


Fig. 14—Demonstration of Metal Flow Produced by Right and Wrong Radii at Base of Rib.

great deal of difficulty. The design looks simple enough, but its effect on the dies is quite disastrous. Note the "whiskers" and rough spots along the edge of the piece.

The die was badly checked and cracked all along the bottom corner of the impression. This condition developed after relatively short use of the die and means, of course, that a new die must be made before additional parts can be produced. The action on the die in this case may be partly due to the explosion of die lubricating oil trapped in the recess, but the principal cause of failure is the wedging action of the metal being forged. Every blow of the hammer drives the metal against the die and exerts a force which tends to split the die apart. The sharper the radius, the more concentrated and severe that force becomes and the sooner failure of the die block takes place. If the design will permit it, ribs and flanges should have full radii for longer die life and ease of filling. Where this is not possible and relatively sharp corners must be obtained, it is necessary

to work out some combination of operations so that the radii are reduced without too much strain on the die block. This always entails additional impressions and increased die expense.

The two sketches in Fig. 14 present still another answer to the question of small radii. This condition is particularly apt to occur when there are ribs on that portion of the forging next to the flash,

Table III
Shrinkage Tolerances for Aluminum Forgings

Length or Width Inches	Shrinkage Tolerance Inch	
	Plus	Minus
1	0.004	0.002
2	0.008	0.004
3	0.012	0.006
4	0.016	0.008
5	0.020	0.010
6	0.024	0.012
For each Additional Inch Add	0.004	0.002
For Example		
18	0.072	0.036
36	0.144	0.072
48	0.192	0.096
60	0.240	0.120

especially in the live die. These ribs will fill before the forging is brought down to size and there is still a certain amount of metal which must be caused to flow past the rib and out into the flash. This metal, of course, moves very rapidly with each blow of the hammer. With a sharp radius the moving metal cannot change direction quickly enough to follow the surface of the forging, but continues to flow straight through the rib and comes out on the other side, thus shearing the rib away from the main body of the forging. This type of defect is aptly known as a "flow through". When the radius is increased as shown on the view to the right, the metal has sufficient time to follow the contour of the forging and no defect is produced.

Shrinkage, which results from the cooling of the metal after forging, causes a dimensional contraction of the forging in all directions. The coefficient of expansion of the various aluminum forging alloys ranges between 0.000011 and 0.000015 inches per inch per degree Fahr. The shrinkage dimensions for the average aluminum forging will come within the plus and minus tolerances given in Table III. However, in special cases the forging will come out of the dies predominantly long. This occurs when the design calls for long forged parts with projections or heavy sections on either end and comparatively thin sections connecting them. During forging,

the lighter middle sections will cool faster, but cannot shrink because the heavy end sections are locked in the die. The forging then comes out of the die at a relatively low temperature and little shrinkage takes place. This type of forging is well illustrated by the large wing support shown in Fig. 15. This intricate forging could be produced satisfactorily, but its use was discontinued because of machining

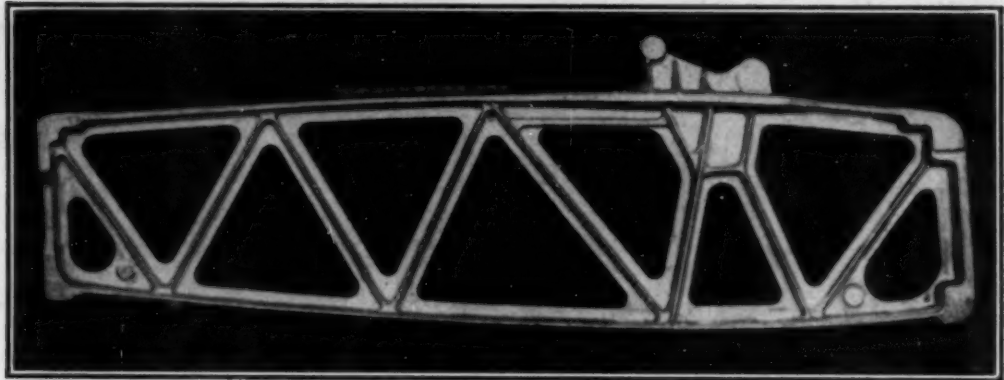


Fig. 15—Large Wing Support Forging. 14S-T Alloy.

and other difficulties. It is advisable to make the projections at one point on such a forging with standard finish allowances and then add increasing amounts of finish to the heavy sections as they occur farther away from that point. Machining layout then should be made from the same point. In this way irregularities in shrinkage are compensated for and there is always sufficient metal provided for machine finishing.

Pockets and recesses in forged parts are formed by corresponding raised sections in the dies. Therefore, pockets should not be too deep and recesses should be as simple as possible in shape. It is recommended that generous fillet radii be provided at the bottom of all pockets and recesses and that the depth of a pocket should not exceed two-thirds of its diameter.

Punch-outs are frequently introduced in hubs and bosses to facilitate the machining and boring of holes. Holes may also be punched in webs and flanges for the purpose of lightening the part or for clearance with some adjacent part. Holes in hubs and bosses are preformed during forging by the introduction of a depression in each side of the part with a web not less than $\frac{1}{8}$ inch thick left in the forging. This web is punched out taking care that the hole should be no larger than the tangent points of the fillet radii. Holes

in webs and flanges are also preformed during forging and can be punched out during the trimming operation. Usually such depressions are flashed and guttered in order to allow excess metal to escape.

Another factor should be mentioned in conjunction with the discussion of forging design because it also has a real effect on die life,

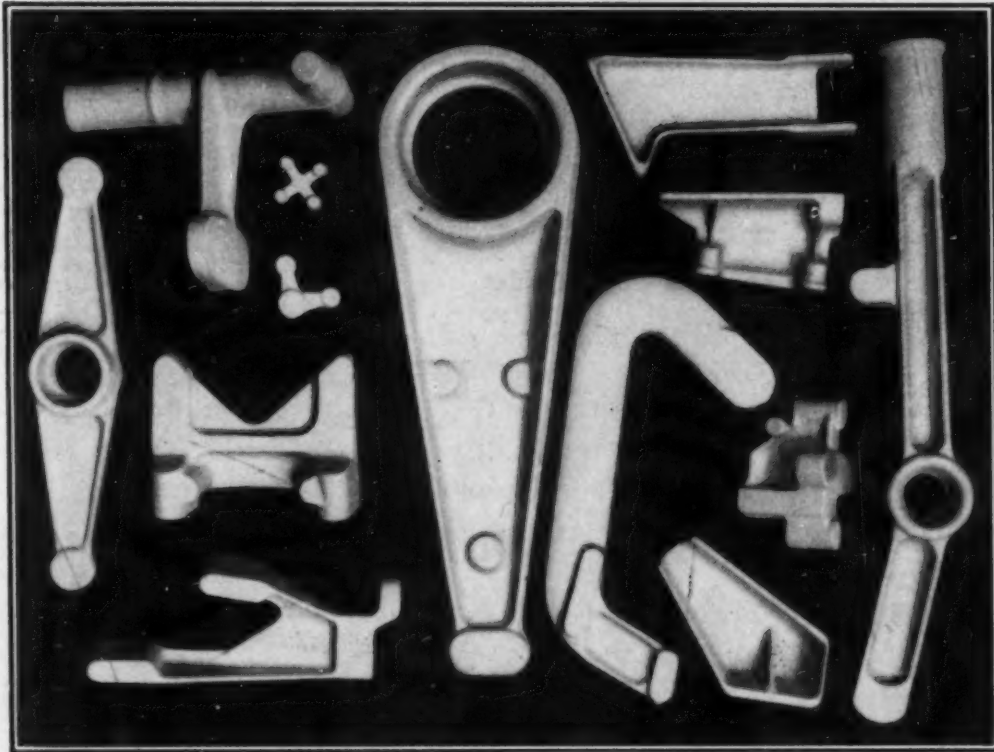


Fig. 16—Representative Group of Miscellaneous Forged Airframe Fittings.

forging quality and forging cost. This factor is the lubrication of the forging dies. This problem is more or less unique to the production of aluminum and magnesium forgings since little or no lubrication is necessary during the forging of other metals.

Probably the most important factor in effective die lubrication is the method of application regardless of the type of lubricant employed. Wherever possible, spray guns should be used to apply the lubricant to the dies. This permits more uniform distribution than either swabs or brushes. It also means that lighter lubricants can be used on all types of forgings, thus reducing the smoke nuisance. Further improvement can be obtained by education of the operators to a more judicious use of the lubricants.

Smooth, polished die impressions are essential to the proper

performance of any lubricant. Where machining marks are still visible, there is a marked tendency for aluminum pick-up on the die. Hot forging lubricants are merely protective films separating aluminum and steel, and irregularities on the surface of the die will tend to break this film down and permit the aluminum part to gall against the steel.

The use of vents in closed dies and closed portions of dies should be given serious attention. Such vents permit the escape of trapped gases resulting from explosion and burning of lubricants and prolong materially the life of the dies.

Forgings made from the correct materials with good design and produced in the right way on the proper type of forging equipment represent the highest form of development of the metal working art. In other words, a forging, in most applications, can accomplish the desired results with less metal than any other type of fabricated metal product. The use of a forging, then, depends upon the value to the user that he can secure by the incorporation of forged parts in his product. In many places a forging may actually be the cheapest product that can be employed, especially in large scale production, because on modern machinery forgings can be produced so rapidly with small dimensional variations, smooth surfaces and resultant savings in finishing time. In cases where forgings are actually more expensive on the basis of total cost in the finished assembly, they may still be desirable, or even necessary, in view of the uniformly high quality of the product.

The aircraft industry has been the backbone of the aluminum forging business for years and probably will continue to be the biggest consumer for forgings for some time to come. In this field the trend toward large complicated forgings will probably continue, as it should. Several years ago it was general practice for aircraft designers to specify small forged fittings which were joined by other types of structural material. Then someone put a group of fittings together and redesigned that group so that a single larger forging could be used in its place. This resulted in higher material cost, but lower over-all construction cost because of the elimination of costly assembly labor. Representative airframe fittings in a wide range of sizes may be seen in Fig. 16. Redesigning has resulted in a steady increase in the size of forgings until an entire wing support such as that shown in Fig. 15 has been produced as a single forging. These changes in the physical dimensions of forged aircraft structural parts

have been made possible only by the use of large forging equipment described in the early part of this paper. With the release of this equipment from war production demands further development of new large airframe structural parts and parts for other application is expected.

Of course, in airplane power plants aluminum forgings have such an outstanding record that their continued use is certain. This use should increase with wider application of the forged cylinder head. With the mechanized setup for the production of this part that is in use today, it will undoubtedly prove more economical than the cast head in addition to the fact that it appears to be a more efficient head in service.

Demands for war production have increased the capacity of the aluminum forging industry to such an extent that a large part of this capacity will be available for uses other than aircraft in the post-war period. Parts that are used continuously and in considerable volume are ideal. Naturally other branches of the transportation industry present the most likely field for development. Such items as railway journal bearings, truck and bus wheels and automotive axle housings are now under consideration. There is the possibility of producing complicated or large parts such as railway car truck frames and pistons for large Diesel engines. Certainly there are many other applications that have not been mentioned here, so that aluminum alloy forgings will have a very intriguing future.

(Some of the alloy compositions and forging practices described above are covered by United States patents owned by Aluminum Company of America.)

DISCUSSION

Written Discussion: By A. L. Rustay, assistant chief metallurgist, Wyman-Gordon Co., Worcester, Mass.

Under the heading "Standard Forging Alloys" some consideration has been given to the corrosion resistance of the alloys as one factor in determining their use in aircraft forgings. Inasmuch as German aircraft use considerable magnesium, which is much more reactive than aluminum, it is doubtful whether the difference between the aluminum alloys with regard to resistance to corrosion warrants any consideration as a factor in determining the choice of a forging alloy.

On pages 159 and 160 the drop hammer test is described. Quantitative data obtained from these tests would help remedy the situation mentioned earlier

in the paper about the dearth of information available in technical literature regarding aluminum forging developments.

Full power, full stroke hydraulic presses, we agree, are certainly desirable, and our experience with them bears out the writer's enthusiasm for this type of equipment.

Again we agree with the writer that proper die design is a necessity in obtaining satisfactory grain flow. The nature of the metal flow in aluminum, which is distinctly different in some respects from the way steel behaves, seems to make it more susceptible than steel to certain types of surface laps as well as interior defects due to a sort of "extrusion pipe".

Fig. 6 shows an interesting structural fitting. It would be of additional interest if a grain flow of the part were shown in view of the unusual forging die design used to make the part.

Fig. 10 emphasizes the directional properties obtained in aluminum forgings. It is a good illustration of the fact that separately forged test bars are of little value to the designer or user of forgings in determining whether or not the parts have adequate properties. As the forging becomes increasingly large or complex, then the conditions under which the test bars and the forgings are made become increasingly divergent. If it is considered desirable to include test bars of some sort as a check on the heat treating practice, it seems more reasonable to use rolled or extruded bar, although again, tests on these separate coupons seem to be a poor substitute for careful checking of furnace temperature controls and adequate testing of the heat treated forgings themselves.

It has been our experience that small laps in steel forgings can weld up in the forging operation, but the associated decarburization which may extend far below the finish line of the forging never disappears. This decarburization of course is not as serious a defect as an actual lap, but steps are always taken to correct the dies to eliminate this condition because it is a potential source of fatigue failure.

In the discussion of advantages of large radii, top of page 174, it might be noted that in addition to the advantages listed, the larger radii tend to minimize stress concentration which is certainly a factor of prime importance to the designer.

Written Discussion: By H. F. Walker, metallurgist, Chevrolet-Muncie Division of General Motors Corp., Muncie, Ind.

The author is to be congratulated for a very interesting and concise paper on Aluminum Alloy Forging. In my opinion, this paper has been so amply handled that little discussion is warranted.

A large part of the text is given to die design and too much emphasis cannot be placed on this phase of the operation. Experience has taught many times over, that success or failure in producing a good forging is largely dependent on forging and/or die design.

One question, prominent in our mind, we would like to ask Mr. Davis—in considering the superior corrosion resistance properties of 17S-T and 14S-W as against 14S-T, is this attributed to lack of precipitation in the case of 17S-T, precipitation in critical areas and size, or some other factor?

Written Discussion: By C. M. Campbell, metallurgist, Chevrolet-Transmission Division of General Motors Corp., Saginaw, Mich.

In Mr. Davis' excellent paper there is no opportunity for criticism; however, several questions arise as to what forging temperatures to use for the various alloys and when to use them.

When forging ingots or material cut from ingots a lower temperature must be used due to condition of cast structure. However, if rolled stock is being forged, a higher temperature can be used.

Regular practice on 14S ingots forged under a 3000-ton press is to use a temperature of 750 degrees Fahr. (400 degrees Cent.). If the forging is to be forged further under a hammer a temperature of 800 to 840 degrees Fahr. (425 to 450 degrees Cent.) can be used depending on the differential between the rough and finished size or spread to be obtained.

The author mentions that the optimum forging temperature of 18S is a little above 800 degrees Fahr. (425 degrees Cent.). However, we have forged thousands of pistons from 18S rolled from bar stock using a temperature of 880 degrees Fahr. (470 degrees Cent.). Bar stock size is 3.5 inches in diameter and finished forging 5.25 inches in diameter. However, if a more intricate forging were being made and the spread were increased it would be necessary to use a lower temperature.

The zinc magnesium alloys are hard to forge, forging temperatures are lower than on the other alloys and splits and ruptures are more prevalent. Die breakage and equipment repairs are excessive and unless the higher physicals are absolutely essential one of the other alloys should be used.

In most cases there is a fairly wide spread between the minimum and maximum temperatures that can be used. The highest forging temperature that can be used without danger of rupturing the material will save dies, equipment and give a more uniform quality forging.

Oral Discussion

S. A. GORDON:¹ Mr. Davis mentioned very lightly the elongation on large forgings. An occasion arose for us to secure several large forgings at one time, and we had noted through a series of tests that the cross-grain elongations varied from about 1.5 per cent to 0.0 per cent and the with-grain elongations varied anywhere from 6.0 to 3.0 per cent. Mr. Davis mentioned that these values (though he did not quote them, I assume that he had them in mind) were not alarming. I would like to have Mr. Davis elucidate to some extent as to what is and is not alarming on elongations of large forgings. If they are not alarming we would certainly be encouraged. If they are, what sort of tests would be suggested to check their integrity in airplane structures?

T. L. FRITZLEN:² Several years ago there was considerable difficulty with blisters on forgings throughout the country, and it was my experience to have been called in on a problem due to the fact that we supplied stock. Quite a few of the blisters were definitely attributed at the time to the stock and heat treatment but there is one question which I would like to ask Mr. Davis his opinion

¹Chief test engineer, Glenn L. Martin Co., Baltimore.

²Chief research metallurgist, Reynolds Metals Co., Glen Cove, N. Y.

on, and that is whether or not the die for forging can be designed in such a manner that the metal, instead of flowing, will shear internally to cause blisters upon subsequent heat treatment.

Author's Reply

Mr. Rustay has presented some very interesting points in his discussion, particularly in his references to the use of magnesium in German-aircraft forgings. However, I do not believe that the employment of magnesium for this purpose necessarily means that we should disregard the resistance to corrosion of various aluminum alloys in selecting the proper material for various applications. In general, for aircraft forgings that alloy providing the highest strength to weight ratio will be employed. However, there have been some applications in the past, particularly where pressed-in bushings are employed, in which it was necessary to select material with the maximum resistance to corrosion. There are other fields for the application of forgings apart from aircraft where these differences in resistance to corrosion are of major importance. This applies especially to the dairy industry, the brewing industry, and the rayon and textile fields in which we should see many post-war applications of aluminum forgings.

I agree with Mr. Rustay that quantitative data from the drop hammer test would have been a very helpful part of this paper, but unfortunately under present conditions it did not appear advisable to release this quantitative information for publication at this time.

I must differ with Mr. Rustay in his conclusions drawn from the tensile properties obtained in different locations in the forging shown in Fig. 10. The results obtained on this forging demonstrate the fact that we cannot rely upon a test specimen cut at random from a forging as an indication of the compliance of such forgings with existing specifications. The variation in properties, particularly elongation, within a single forging is much greater than the variation that would be expected from one forging to another and, therefore, we are brought to rely upon separately forged test specimens as a more reliable indication of the effectiveness of heat treating practice. If, in some special cases, tests from the forgings themselves are desired, these results indicate the necessity of determining a specific location from which the test bars shall be taken, and the establishment of an individual specification for this part indicating the properties that should be obtained from such a test.

I appreciate Mr. Campbell's remarks, and he has added some information on forging temperatures that is interesting and valuable. His remarks emphasized the relationship between forging equipment, die design, and forging temperatures. As he has indicated, the maximum forging temperature for any alloy appears to be related to the rate of movement of the metal during forging as well as the type of blow that produces the metal movement. This is a field that has not been covered as thoroughly as it should be, and further work along these lines should increase the possibilities for forging the various aluminum alloys.

In answering Mr. Walker's question, I would like to point out that it is not generally true that 17S-T and 14S-W have corrosion resistance properties superior to 14S-T. Under certain conditions some one of these alloys may be superior to the other two, while under other conditions, another alloy will be

slightly better. However, for all practical purposes we can consider that the resistance to corrosion of all three of these materials is essentially equal and that with the proper heat treatment, all of them will possess very good corrosion resistance.

In response to Mr. Gordon's question in regard to elongation, I think the evidence of the effect of these reduced elongations is found in the serviceability of the forgings containing them. For instance, in the forging that was shown in Fig. 10 you will notice that the bars vertical to the parting line in the upper right hand corner, numbered 7 and 8, each show elongations of 2.5 per cent. Yet this forging is performing very satisfactorily in one of our larger bombers and has been in use for several years.

Referring to blisters mentioned by Mr. Fritzlen, there are, of course, many causes for blisters of which the quality of the forging stock is one. Dies can be improperly designed and the forging can be worked in such a way that it is sheared internally. Such a condition, however, is much more apt to occur from improper lubrication of the forging die while the forgings are being made, causing the outer surface of the metal to adhere to the die surface, while the inner portion of the metal flows past, shearing a thin film of metal on the exterior of the forging and permitting the entrance of air which during heat treatment causes blisters. Other factors are also important in analyzing the cause of blisters on forgings. There are too many of these factors to go into detail regarding them at this time.

THE PROPERTIES OF ALUMINUM ALLOYS MELTED IN AN INDUCTION HEATED CRUCIBLE FURNACE

BY JAMES W. POYNTER

Abstract

Physical properties of cast and heat treated test bars of aluminum alloys conforming to Specifications AN-QQ-A-390 (Alcoa 195), AN-QQ-A-376 (Alcoa 355), and AN-QQ-A-394 (Alcoa 356), were determined. The metal from which these bars were cast was melted in a clay graphite crucible heated by induced high frequency electric currents. No fluxing treatment was used. The properties obtained were in excess of the minimum values required by the specifications. These properties and the compositions of the alloys were substantially unchanged by remelting and recasting for as many as seven times. These results were compared with the results obtained on test bars produced in routine foundry practice, which included fluxing with chlorine gas. No significant difference was found in the physical properties, soundness, or microscopic structure. In an emergency such as a shortage of chlorine gas, castings of good quality and strength could be produced in induction furnaces.

MOLTEN aluminum reacts with water vapor to form aluminum oxide and hydrogen gas. The aluminum oxide formed by this reaction is an inactive compound. Since aluminum and its alloys in the liquid state will absorb large quantities of hydrogen,¹ this hydrogen is dissolved. Upon solidification most of this gas is liberated, producing gas cavities of various sizes which Sachs and Van Horn² state "represent one of the most frequent defects in castings". The water vapor for this reaction may be present in the atmosphere in the room, in the products of combustion in fuel-fired furnaces, or as water absorbed on the solid metal.

¹L. L. Bircumshaw, "The Solubility of Hydrogen in Molten Aluminum", *Transactions, Faraday Society*, Vol. 31, 1935, p. 1439-1443.

²G. Sachs and K. R. Van Horn, "Practical Metallurgy", published by the American Society for Metals, 1941, p. 190.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, James W. Poynter, is connected with the Materials Laboratory, Army Air Forces, Materiel Command, Wright Field, Dayton, Ohio. Manuscript received May 24, 1944.

With a closed crucible and electric power as a source of heat, no water vapor from the products of combustion would be present and the water vapor from the atmosphere and absorbed on the solid metal could be minimized. Under these conditions, it would be expected that satisfactory physical properties of aluminum alloy castings could be obtained without the use of any of the fluxing methods generally used to remove hydrogen.

In this investigation, the physical properties of aluminum casting alloys melted in a crucible furnace heated by induced high frequency electric currents were determined and compared with the properties of the same alloys melted in gas-fired crucible furnaces.

EXPERIMENTAL METHOD

Three casting alloys, Specification AN-QQ-A-390 (Alcoa 195), Specification AN-QQ-A-376 (Alcoa 355), and Specification AN-QQ-A-394 (Alcoa 356), were selected. Three hundred and fifty-pound batches of these alloys were made, using aluminum ingot and the appropriate intermediate alloys. This charge was melted in a gas-fired crucible furnace and was cast into pigs, using iron molds.

For the first induction melt of an alloy, 35 pounds of these pigs were weighed out for the charge. As much of the charge as possible was placed in the clay graphite crucible and the power turned on. As the melting of the metal made space available, the rest of the charge was added to the crucible. The completely molten metal was then transferred to a preheated crucible held in a hand shank and 2 TBG molds (Fig. 11-B, Specification QQ-M-151) poured at 1250 degrees Fahr. (675 degrees Cent.).

The remainder of the metal was re-pigged. The subsequent melts were made using the gates, risers and pigs from the previous melts. The melting crucible was kept covered as much of the time as possible. The portion of the charge which could not be placed in the crucible at the beginning of a melt was laid on the cover so that it would be dry and preheated before placing in the crucible.

A special technique was developed to control the pouring temperature. Approximately 4.5 pounds of metal (usually two gates) were kept back when the rest of the metal was charged. When no more solid metal was visible in the crucible, this 4.5 pounds of metal, which had been preheated on the top of the furnace, was added and the power supply shut off after 2 minutes. The heat stored in the

crucible, insulation, etc., was usually sufficient to bring the metal up to the desired pouring temperature. An immersion type thermocouple was used to measure the actual temperatures, both in the furnace and in the pouring crucible.

Since the clay graphite crucible used was a conductor of electricity, the molten metal was not appreciably stirred by the action of the high frequency current. As a tendency for metal in the mushy state to collect at the bottom of the crucible was noted, the metal was stirred thoroughly with a graphite rod a few minutes before pouring. The test bars were solution heat treated, cold-water-quenched, and aged in accordance with Specification AN-QQ-H-186.

RESULTS

The physical properties obtained on these test bars appreciably exceeded the specified minimum in all cases except that the elongation obtained on the 355 alloy test bars was only slightly higher than the specified value (Tables I, II and III.)

The physical properties were not appreciably changed by remelting for as many as seven times (Tables I, II and III.) The average of the results obtained on the test bars from all seven melts was therefore taken as representative of the properties obtained. Except for a slight iron pickup, the composition of the alloys was unchanged by the repeated melting operations.

The power consumption was 30 KVA (the rated capacity of the converter) when the line voltage was 215. Drops in the line voltage caused definite decreases in the power consumption. While variations in the weight of the charge did not affect the power input, the smaller charges were naturally melted down more rapidly than the larger ones. The melting time varied from about 35 minutes for an 18-pound charge to 70 minutes for a 35-pound charge.

In routine Wright Field foundry operation, a 350-pound melt of an alloy composition was cast into ingots. Some of these ingots were then remelted in a small gas-fired crucible furnace fluxed with chlorine gas and cast. As a control, one or two molds of test bars were cast and heat treated with each lot of castings. To better evaluate the effect of the induction melting on the physical properties of the metal, the results obtained on the test bars cast from the metal melted in the induction furnace were compared with the results obtained on the control test bars cast in routine production. Since all the test bar molds were

Table I
Effect of Remelting on the Physical Properties of 195 Aluminum Alloy Test Bars

Remelt	Yield Strength psi.	Tensile Strength psi.	Elongation % in 2 inches	Brinell Hardness
First	31,500	45,600	6.5	89
Second	29,600	43,800	6.0	91
Third	31,000	44,800	6.0	93
Fourth	29,600	42,600	6.0	84
Fifth	28,100	41,100	5.5	84
Sixth	30,700	41,700	4.5	88
Seventh	31,800	42,800	4.5	84
Specification AN-QQ-A-390 (minimum)	20,000	32,000	3.0	..

Table II
Effect of Remelting on the Physical Properties of 355 Aluminum Alloy Test Bars

Remelt	Yield Strength psi.	Tensile Strength psi.	Elongation % in 2 inches	Brinell Hardness
First	33,800	40,100	2.5	97
Second	34,400	40,600	2.0	100
Third	34,000	39,900	2.0	100
Fourth	33,800	40,000	2.0	99
Fifth	34,600	40,700	2.5	100
Sixth	33,700	39,500	2.0	98
Seventh	33,800	40,000	2.0	97
Specification AN-QQ-A-376 (minimum)	20,000	32,000	2.0	..

Table III
Effect of Remelting on the Physical Properties of 356 Aluminum Alloy

Remelt	Yield Strength psi.	Tensile Strength psi.	Elongation % in 2 inches	Brinell Hardness
First	21,700	34,100	6.5	75
Second	21,700	33,300	5.5	73
Third	21,600	33,900	6.0	74
Fourth	21,400	32,800	5.5	73
Fifth	21,500	33,500	6.5	74
Sixth	22,000	34,000	7.0	74
Seventh	22,000	33,200	6.0	73
Specification AN-QQ-A-394 (minimum)	20,000	30,000	3.0	..

made by the same foundry crew, using the same patterns, gating and sand, any variation in the properties could be attributed to differences in the melting practice or in the heat treatment. In making this comparison, two sets of results were taken from the foundry records for each alloy. One set was the result of all the tension tests made on control bars which had been cast from the 350-pound melt of metal from which the ingots for the induction furnace melt were taken. The other set was the result of all the tension tests made on control test bars from another recent 350-pound melt of the same alloy.

Table IV
Chemical Composition of Melts Used for Comparison of Mechanical Properties

Furnace Production	Induction Experimental	Gas Routine Foundry	Gas Routine Foundry	Specification Requirements
	Specification 7706	AN-QQ-A-390 7391	(195) Alloy 7054	
Melt No.	7706	7391	7054	
Copper	4.40	4.40	3.79	4.0—5.0
Silicon	0.75	0.75	0.65	1.5 maximum
Iron	0.45	0.45	0.25	1.0 maximum
Titanium	0.18	0.18	0.19	0.2 maximum
	Specification 7788	AN-QQ-A-376 7748	(355) Alloy 7608	
Melt No.	7788	7748	7608	
Silicon	5.19	5.19	4.53	4.5—5.5
Copper	1.42	1.42	1.17	1.0—1.5
Magnesium	0.56	0.56	0.51	0.4—0.6
Iron	0.50	0.50	0.43	0.5 maximum
Titanium	0.16	0.16	0.16	0.2 maximum
	Specification 7730	AN-QQ-A-394 7374	(356) Alloy 6316	
Melt No.	7730	7374	6316	
Silicon	6.67	6.67	7.06	6.5—7.5
Magnesium	0.37	0.37	0.35	0.2—0.4
Iron	0.43	0.43	0.29	0.6
Copper	0.03	0.03	0.10	0.2
Titanium	0.19	0.19	0.18	0.2

Note: All analyses made on drillings taken from three ingots and mixed.

The melt numbers and the composition of the melts, as determined on the cast ingot, are listed in Table IV.

The comparison of the physical properties of the induction furnace melted 195 alloy (Melt 7706) test bars with those of the gas-fired furnace melted control bars (Melts 7054 and 7391), showed that the yield strength of Melt 7706 was higher, the elongation lower and the tensile strength approximately the same (Table V and Fig. 1.) These differences are not considered significant in regard to melting practice, since the Melt 7706 test bars had been aged for a longer time than many of the bars from Melts 7054 and 7391. Although both aging treatments were in conformance with Specification AN-QQ-H-186, the longer time aging used for Melt 7706 would produce a higher yield strength with a corresponding decrease in the elongation. All of the values were appreciably in excess of the minimum specified in Specification AN-QQ-A-390.

The average yield and tensile strength values found for the induction furnace melted 355 alloy test bars (Melt 7788) were higher than the averages for the control test bars (Melts 7608 and 7748) melted in the gas-fired furnace (Table VI and Fig. 1.) Although the maximum values for the yield and tensile strengths were about the same for all three melts, the minimum values for Melts 7608 and 7748 were considerably lower than the minimums for Melt 7788. The observed differences in the average ductility were considered to

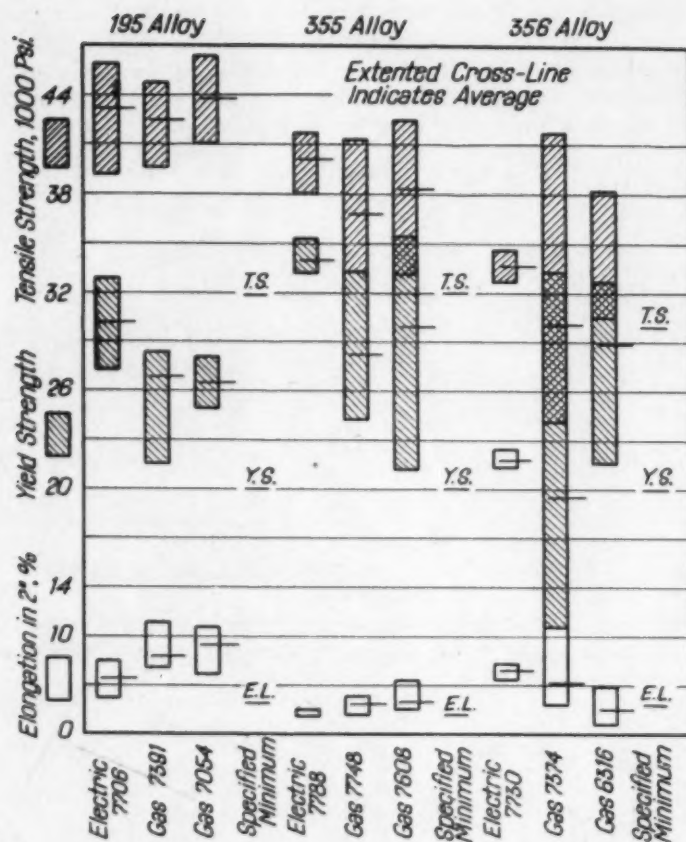


Fig. 1—Comparison of Ranges of Physical Properties Obtained on Aluminum Alloys Melted in Electric and in Gas Furnaces.

Table V
Comparison of Mechanical Properties Obtained on 195 Alloy Test Bars Cast From Metal Melted in Gas and Electric Furnaces

	No. of Values in Average	Yield Strength psi.	Tensile Strength psi.	Elongation, % in 2 inches	Brinell Hardness
Induction Melt 7706	20				
Maximum		32,800	45,900	7.5	93
Minimum		27,200	39,100	4.0	83
Average		30,100	43,300	5.5	88
Gas Melt 7391	21				
Maximum		28,300	44,700	11.5	85
Minimum		21,300	39,400	7.0	78
Average		26,700	42,400	7.9	81
Gas Melt 7054	30				
Maximum		28,000	46,400	11.0	87
Minimum		24,900	41,000	6.0	79
Average		26,500	43,700	9.0	84
Specification AN-QQ-A-390		20,000 min.	32,000 min.	3.0 min.	..

be due, at least in part, to the error inherent in measuring small elongations. All values met or exceeded the requirements of Specification AN-QQ-A-376.

Table VI
Comparison of Mechanical Properties Obtained on 355 Alloy
Test Bars Cast From Metal Melted in Gas and Electric Furnaces

	No. of Values in Average	Yield Strength psi.	Tensile Strength psi.	Elongation, % in 2 inches	Brinell Hardness
Electric Melt 7788	21				
Maximum		35,300	41,700	2.5	102
Minimum		33,100	38,100	2.0	96
Average		34,000	40,100	2.2	99
Gas Melt 7748	33				
Maximum		33,200	41,300	4.0	98
Minimum		24,200	32,800	2.0	83
Average		28,400	36,900	3.0	90
Gas Melt 7608	27				
Maximum		35,400	42,600	5.5	95
Minimum		21,000	33,200	2.5	75
Average		29,900	38,500	3.2	87
Specification		20,000	32,000	2.0	..
AN-QQ-A-376		min.	min.	min.	

Table VII
Comparison of Mechanical Properties Obtained on 356 Alloy
Test Bars Cast From Metal Melted in Gas and Electric Furnaces

	No. of Values in Average	Yield Strength psi.	Tensile Strength psi.	Elongation, % in 2 inches	Brinell Hardness
Electric Melt 7730	20				
Maximum		22,500	34,500	7.0	76
Minimum		21,400	32,600	5.5	72
Average		21,800	33,600	6.2	74
Gas Melt 6316	75				
Maximum		33,100	41,700	11.0	98
Minimum		11,500	24,100	3.0	50
Average		19,600	30,000	5.1	67
Gas Melt 7374	22				
Maximum		32,600	38,100	5.0	95
Minimum		21,600	30,300	1.0	73
Average		29,000	35,000	2.5	88
Specification		20,000	30,000	3.0	..
AN-QQ-A-394		min.	min.	min.	

The average properties obtained on Melt 7730 (induction furnace melted 356 alloy) were appreciably better than the average obtained on gas-fired furnace Melt 7374 (Table VII and Fig. 1.) However, the maximum individual yield strength value for Melt 7374 was within 500 psi. of the average tensile of Melt 7730, while the minimum individual yield and tensile values were much lower than the minimums specified in Specification AN-QQ-A-394. The average tensile strength of Melt 7374 just met the specified minimum and the average yield strength was slightly less than specified value. The average tensile value for gas-fired Melt 6316 was higher than Melt 7734, the average yield strength was much higher and the average elongation lower. These differences were due to the way in which the melts responded to the same aging treatment.

The 356 alloy, Melt 7374, from which the ingots for induction melting were taken, was an "off" heat. By this is meant that, while the composition is within the specified limits, the minimum physical properties were not always developed by the usual heat treatment. Some castings of Melt 7374 had to be given a second solution treatment at a higher temperature and for a longer time than the standard in order to meet the minimum specification requirements. The physical properties of other castings from the same melt, as judged by results obtained on test bars, were exceptionally high. This anomalous behavior was responsible for the very wide range over which the test bar results were distributed. The overall average, however, barely met the specified minimum. While the average properties of the induction furnace Melt 7730 were definitely superior to those of the parent Melt 7374, and exceeded the specification requirements, the average tensile strength and yield strength were not as good as those of Melt 6316 (gas-fired furnace). Since Melt 7730 showed a higher ductility, this difference was attributed to the sluggish response to heat treatment of Melt 7730 and to possible differences in the artificial aging treatment.

The range of values for the physical properties was much less for the induction melted 355 and 356 alloys than for the gas furnace melted alloys. This better uniformity was attributed to the fact that the test bars of the induction melted alloys were heat treated in the same furnace charge while the gas furnace melted test bars were heat treated in a number of different furnace charges. Little difference between the uniformities of the gas and induction furnace melted 195 alloys was noted. This may be due to the lesser sensitivity of the 195 alloy composition to variations in the solution and aging treatments.

Radiographic examination was made of all bars cast from metal melted in the induction furnace. A very few of them contained obvious defects (pieces of refractory, etc.) and were discarded. No difference in quality and soundness was detected between the rest of these bars and bars cast from metal melted in gas-fired crucible furnaces.

The microscopic structure of these induction furnace melts was normal and could not be differentiated from typical gas-fired crucible furnace melts.

A number of processes are used commercially to remove hydrogen from aluminum. Some of these consist in holding the metal for

a long period slightly above the melting point; allowing the metal to solidify slowly followed by a rapid remelting; flushing an inert gas, such as nitrogen, through the liquid metal; flushing gaseous chlorine through the liquid metal; or treating the metal with volatile chlorides.

The test results indicate that such precautions are not necessary for metal melted in an induction furnace, provided a clean ingot relatively free from hydrogen is used, since adequate physical properties and soundness are obtained without these special treatments. This would be expected since no products of combustion of a fuel are present and since the metal was thoroughly dried before melting. It is true that water vapor in the air is present but its deleterious effects would be at the minimum since the crucible is covered, thus preventing free access of the atmosphere with the attending water vapor to the molten metal.

Of the commercial processes previously mentioned for removing dissolved hydrogen gas from molten aluminum alloy, the use of chlorine gas is quite prevalent. While adequate amounts of this gas are now available, a critical shortage such as occurred earlier in the War might make the continued use of chlorine impossible. In such an emergency, castings of good quality and strength could be produced in induction furnaces. Obviously, furnaces with 40-pound capacity could not adequately meet the demand, but induction furnaces are now being built with a melting capacity of 1250 pounds of aluminum alloy per hour.³ A furnace of this capacity is rated at 250 KW.

CONCLUSIONS

Test bars of 195, 355, and 356 aluminum alloys cast from metal melted in a high frequency induction furnace had physical properties in excess of the minimum values specified in the ANA Specifications AN-QQ-A-390, AN-QQ-A-376, and AN-QQ-A-394, respectively.

The compositions of the alloys and the physical properties were substantially unchanged by repeated remelting and recasting.

The physical properties, soundness and microscopic structure of test bars cast from metal melted in the induction furnace without the use of chlorine gas or other fluxing methods were equal to those of test bars cast from metal which was melted in a gas-fired furnace and treated with chlorine gas.

³Manuel Tama, "Induction Furnace for Electric Melting of Aluminum", METAL PROGRESS, Vol. 44, 1943, p. 968B.

ACKNOWLEDGMENTS

The author wishes to thank J. B. Johnson, Chief, Materials Laboratory, and R. R. Kennedy, Chief, Metallurgical Branch, Engineering Division, for their interest and suggestions in this project and the members of the Materials Laboratory staff for their co-operation in casting and testing the tensile specimens.

DISCUSSION

WALTER BONSAK:⁴ I have read this paper with considerable interest. The author certainly did a nice job. It is especially interesting to a smelter to have confirmation by investigators that aluminum alloys do not have to deteriorate when proper care is taken during repeated remelting.

However, I have a few questions which I would like to have answered. In the chart giving the average physical properties the maximum, minimum and average are listed for tensile strength, yield strength and elongation. We find maximum tensile and yield strength listed with maximum elongation. However, to analyze the large variations occurring in the two magnesium-bearing alloys, it seems to be of importance to know whether the maximum yield strength was always found on test bars having also the maximum elongation and vice versa. If this is the case, an explanation is hard to find for the variations in yield strength and elongation. If, however, the high yield strength is coupled to low elongation and the low yield strength to high elongation, then it is possible to account for the variations by varying magnesium content. Magnesium is lost during remelting and must be compensated for. The loss is not uniform but may vary from melt to melt. This leads to the second question: Has the magnesium content of these alloys been adjusted each time and is there any connection between the magnesium content variations and the variations in physical properties? Losses in sodium and the effectiveness of titanium might also cause variations in these silicon alloys. Have these elements been considered as a basis for an explanation of the variation?

Author's Closure

To prevent any misinterpretation of the results given in Tables V, VI and VII, it should be pointed out that the values given for the maximum yield strength, tensile strength and elongation were not generally obtained on the same test bar.

In reply to Mr. Bonsack's question, the actual elongation values obtained on the 355 and 356 alloy test bars which had maximum and minimum yield strength are given in Table VIII. It will be noted that low, but not necessarily the minimum, ductility is generally found on bars with the maximum yield strength and that high, but not necessarily the maximum, ductility is generally obtained on bars with the minimum yield strength.

⁴Director of Laboratories, The National Smelting Co., Cleveland.

Analyses of the induction furnace metal before the first melt and after the seventh remelt showed no change in either the magnesium or the titanium content.

Since at least 50 per cent of the charge for each routine foundry melt was composed of ingot from a lot analyzed for conformance with the specification, no attempt was made to adjust the magnesium content. Previous investigations have shown that only a slight magnesium loss resulted with the melting equipment and foundry practice used in these tests.

Table VIII
Ductility of Test Bars Showing Maximum and Minimum Yield Strengths

355 Alloy	
Melt 7788	Both the maximum (35,300 psi.) and minimum (33,100 psi.) yield strengths obtained on bars with 2.5 per cent elongation.
Melt 7748	Maximum yield strength (33,200 psi.) on bar with 2.5 per cent elongation.
	Minimum yield strength (24,200 psi.) on bar with 3.5 per cent elongation.
Melt 7608	Maximum yield strength (35,400 psi.) on bar with 2.5 per cent elongation.
	Minimum yield strength (21,000 psi.) on bars with 5.5, 5.5, and 5.0 per cent elongation.
356 Alloy	
Melt 7730	Maximum yield strength (22,500 psi.) on bar with 6.5 per cent elongation.
	Minimum yield (21,400 psi.) on bars with 6.0 and 6.5 per cent elongation.
Melt 7374	Maximum yield (33,100 psi.) on bar with 4 per cent elongation. Minimum yield (11,500 psi.) on bar with 9 per cent elongation.
Melt 6316	Maximum yield strength (32,600 psi.) on bar with 2 per cent elongation.
	Minimum yield strength (21,800 psi.) on bar with 5 per cent elongation.

In conformance with paragraph F-6a (2) of Specification AN-QQ-A-364 which requires that the pouring temperature of the control test bars be within 20 degrees Fahr. of that of the castings they represent, the test bars were poured at temperatures ranging between 1250 and 1375 degrees Fahr. (675 and 745 degrees Cent.). Other tests have demonstrated that increases in the pouring temperature decrease the physical properties obtained.

It is believed that the slight variations in the time and temperature of the aging treatment and the differences in response to aging as previously discussed, together with these differences in pouring temperatures, are the primary factor causing the scatter observed in the foundry melts of the 355 and 356 alloys.

Sodium was neither added to nor determined in these alloys.

MAGNESIUM SHEET

BY P. T. STROUP, G. F. SAGER, AND J. B. WEST

Abstract

A brief discussion is presented on the relation of mechanical properties of magnesium sheet to aluminum content, zinc content, and cold work. Improved resistance to corrosion was shown to result from a decrease in iron content. Stress corrosion cracking was overcome by means of magclad sheet. The essential procedures for forming and joining operations are described. The influence of both the favorable and unfavorable characteristics on commercial applications is reviewed.

INTRODUCTION

THE recent expansion of magnesium production has prompted aircraft manufacturers and others to investigate applications in which sheet of this light metal could be used. In this connection, it was necessary to increase facilities for producing sheet and to obtain much information of a technical nature which would be helpful in the design and construction of products. The present paper briefly describes the manufacture of magnesium sheet and discusses the effect of composition on mechanical properties and resistance to corrosion. The results of some experimental work on stress corrosion cracking are included. Finally some consideration is given to the forming and joining characteristics.

SHEET FABRICATION

In the manufacture of magnesium alloy sheet, the melting charge may comprise metal from several different sources, including virgin ingot which is alloyed during the remelting, prealloyed ingot, scrap and secondary. An excellent description of melting, alloying, and

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. Of the authors, P. T. Stroup is chief and G. F. Sager is research metallurgist, process metallurgy division, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa., and J. B. West is engineer in the jobbing division of the Aluminum Company of America, New Kensington, Pa. Manuscript received July 19, 1944.

refining practices has been presented by C. E. Nelson (1).¹ For sheet fabrication the magnesium alloys are cast into ingots which are either rolled directly or extruded into slab which is then rolled. Prior to rolling, the cast ingots or extruded slabs are preheated, and then hot-rolled to a thickness depending on the final gage desired and on the success of keeping the metal hot enough to continue rolling. These hot mill slabs are cleaned if necessary by etching and mechanical means, annealed and then subjected to alternate stages of cold rolling and intermediate annealing to reach the final gage. Since the amount of cold rolling which magnesium alloys will permit is rather limited, the cold rolling imposed between intermediate anneals ranges between 10 and 25 per cent reduction in commercial operations. The cold-rolled sheet may then be annealed and is usually given a chrome pickle coating before shipping or storage.

The structure of magnesium sheet differs from that of the more commonly known sheet metals—iron, copper and aluminum—by reason of its hexagonal crystal structure and the ease with which twinning occurs. In these two latter respects, it is similar to zinc. The significance of the crystal structure of magnesium and its relation to the behavior on rolling has been considered by Schmid and Wassermann (2), Caglioti and Sachs (3), Schiebold and Siebel (4), Hanawalt (5), McDonald (6) and Bakarian (7). The deformation of magnesium on cold rolling is usually explained by slip along the basal plane with consequent rotation of this plane into the surface of the sheet. It is considered probable that twinning occurs during rolling operations. As the temperature is raised, the mechanism changes and translation on the pyramidal planes has been observed at temperatures above 250 degrees Cent. (7).

COMPOSITION AND PROPERTIES

The published information on the effect of alloying additions on magnesium is considerable for cast and extruded forms but rather meager for cold-worked material. The most frequent and important alloying addition from a standpoint of strength is aluminum.

A series of cold-rolled sheet with different aluminum contents was prepared from small cast ingots by preheating, hot rolling, annealing and finally cold rolling 15 per cent to a gage of 0.064 inch. The tensile properties of these alloys in the transverse direc-

¹The figures appearing in parentheses pertain to the references appended to this paper.

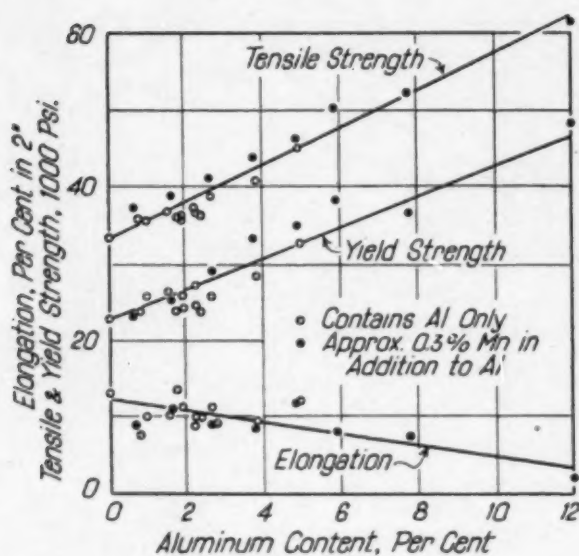


Fig. 1—Effect of Aluminum on Tensile Properties of Cold-Rolled Magnesium Sheet. (15 Per Cent Reduction).

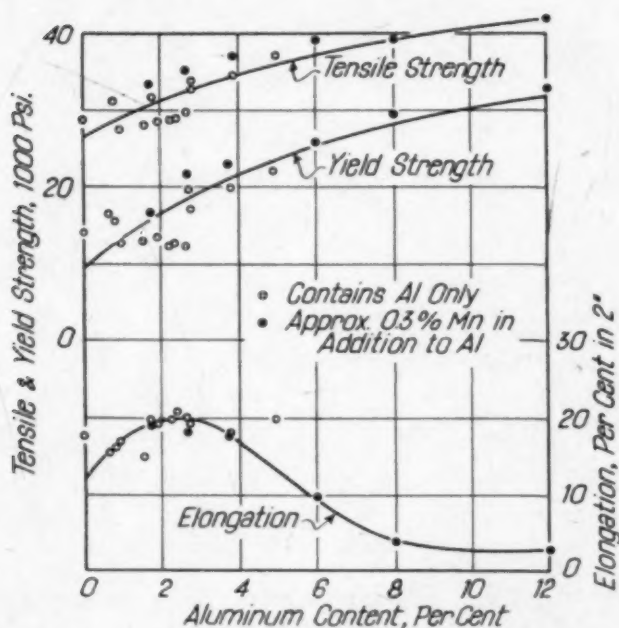


Fig. 2—Effect of Aluminum on Tensile Properties of Annealed Magnesium Sheet. (2 Hours at 650 Degrees Fahr.).

tion in the cold-rolled temper are shown in Fig. 1 and after annealing in Fig. 2. The results in Fig. 1 also include alloys containing 0.3 per cent manganese in addition to the aluminum contents. The addition of manganese had little effect on tensile or yield strength which increased as straight line functions of the aluminum content.

In Figs. 3 and 4, these results are compared with previous re-

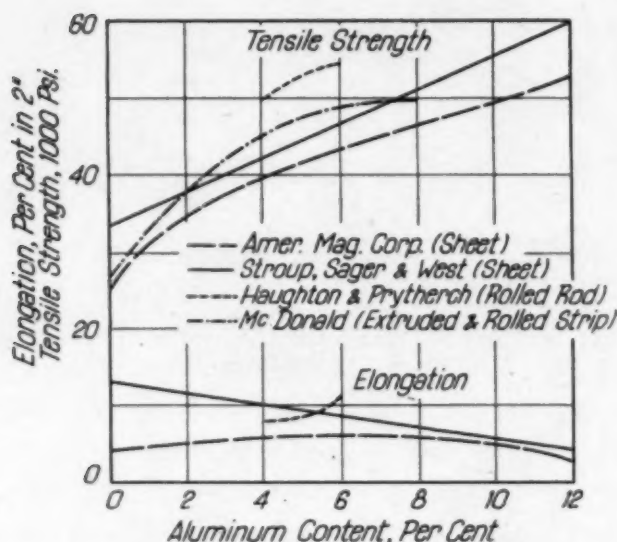


Fig. 3—Effect of Aluminum Content on Tensile Strength and Elongation of Cold-Rolled Material.

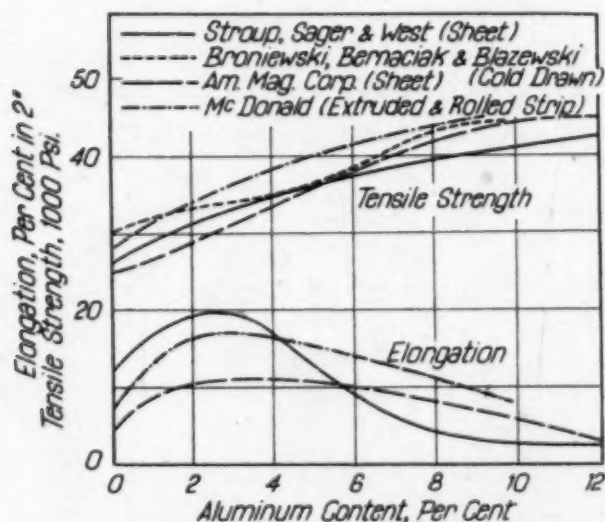


Fig. 4—Effect of Aluminum Content on Tensile Strength and Elongation of Annealed Material.

sults reported in the 1923 edition of the Magnesium Handbook published by the American Magnesium Corporation (8), Haughton and Prytherch (9), Broniewski, Bernaciak, and Blazewski (10), and McDonald (11). Considering the various methods of fabrication of these different investigators it is rather surprising that the results are in such good agreement. In Fig. 4 the elongation values reported by McDonald were maximum and not average values.

Another series of sheet was similarly prepared with several zinc contents and the tensile properties obtained on transverse specimens of these alloys in the cold-rolled temper are shown in Fig. 5

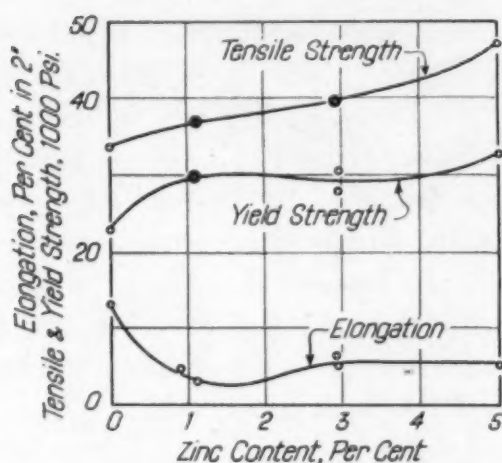


Fig. 5—Effect of Zinc on Tensile Properties of Cold-Rolled Magnesium Sheet. (15 Per Cent Reduction).

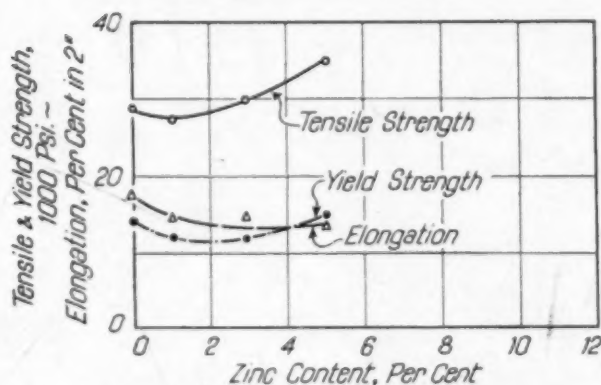


Fig. 6—Effect of Zinc Content on Tensile Properties of Annealed Material.

and for the annealed temper in Fig. 6. The low value obtained for 2 per cent zinc in the rolled temper agrees with that reported by Haughton and Prytherch, while the tensile strength values are similar to those reported by Haughton and Prytherch and by McDonald.

In addition to aluminum and zinc, McDonald reported on the tensile properties of sheet of binary alloys with antimony, bismuth, cadmium, calcium, cerium, copper, gallium, lead, nickel, silver, thallium, thorium and tin.

There is little interest in pure magnesium as sheet except for special purposes but a number of alloy compositions are of interest commercially. The most widely used alloy is one containing 1.5 per cent manganese, which has outstanding welding characteristics with fair forming properties and freedom from stress corrosion cracking. The resistance to corrosion of this alloy is affected little by the iron content. A modification of this alloy containing 0.5 per cent cerium,

in addition to the manganese (Elektron AM537) has been used in Europe to give a finer grain with better drawing and working characteristics as described by Altwicker and Rosenkranz (11a). The addition of a few tenths per cent calcium to the 1.5 per cent manganese alloy has been described by Bulian (12) as resulting in substantial grain refinement with consequent increase in yield strength. The addition of 0.1 to 0.3 per cent calcium to sheet caused a substantial improvement in the cold working characteristics, and had no effect on the resistance to corrosion or stress corrosion or welding properties of the sheet to which it was added.

Table 1
Nominal Compositions and Typical Mechanical Properties of Commercial Sheet Alloys

Mazlo Alloy No.	Composition			Condition	Tensile Properties				Compressive Y. S. (psi.) (X1000)	Shear Strength (psi.) (X1000)	Endurance Limit (psi.) (X1000)	Brinell Hardness	Rockwell Hardness
	Al Per Cent	Zn Per Cent	Mn Per Cent		T. S. (psi.) (X1000)	Y. S. (psi.) (X1000)	El. (Per Cent in 2 inches)						
AM3S-O	1.5	Annealed	32	17	16	15	19	8	44	E-54	
AM3S-H	1.5	Hard Rolled	36	27	9	22	20	10.5	52	E-68	
AM-C52S-O	3	1	0.2	Annealed	38	25	18	16	21	11	54	E-69	
AM-C52S-H	3	1	0.2	Hard Rolled	46	34	10	33	21	11.5	71	E-86	
AM-C57S-O	6.5	1	0.2	Annealed	42	26	10	17	20	12.5	56	E-73	
AM-C57S-H	6.5	1	0.2	Hard Rolled	50	35	6	29	22	...	74	E-89	

Notes: (1) Mechanical properties were obtained on A.S.T.M. specimens. The values given are typical values from which variations must be expected in practice.

(2) Yield strength is the stress corresponding to a 0.2 per cent deviation of the stress-strain curve from the modulus line.

(3) Endurance limits are based on 500 million cycles of completely reversed stress. A repeated flexure type of machine with a cantilever-beam specimen was employed for the tests.

(4) The compressive yield strength values are based on very limited data.

Several sheet alloys containing aluminum by itself or in combination with zinc have been used with the addition of a few tenths per cent manganese to improve the resistance to corrosion. The early so-called high strength magnesium alloys contained 4 to 6 per cent aluminum with 0.3 per cent manganese and no zinc. The zinc was omitted in order to improve the hot rolling characteristics of the alloy. As experience was gained in rolling, zinc was included in these alloys because of its desirable characteristics in giving an increased resistance to corrosion and better cold rolling and forming characteristics. At present two of these alloys are of interest, one of which contains 3 per cent aluminum, 1 per cent zinc, 0.2 per cent manganese and the other contains 6.5 per cent aluminum, 0.7 per cent zinc, 0.2 per cent manganese. Alloys with still higher aluminum

contents are too difficult to fabricate into sheet for commercial production. The compositions and typical properties of the present commercial sheet alloys in both the annealed and hard-rolled tempers are shown in Table I. The alloy designations are those of the American Magnesium Corporation.

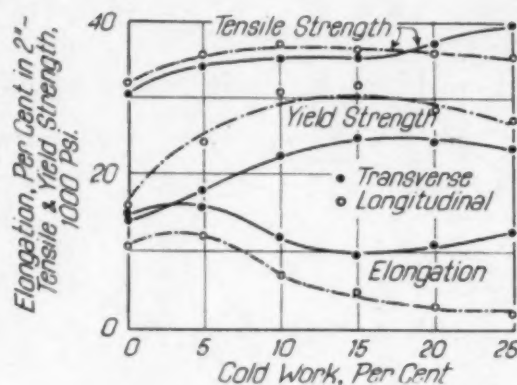


Fig. 7—Effect of Cold Work on Tensile Properties of AM3S.

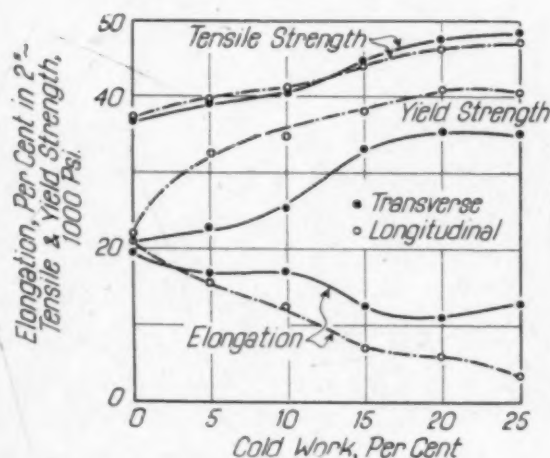


Fig. 8—Effect of Cold Work on Tensile Properties of AM-C52S.

The strength of magnesium sheet may be increased by cold working in addition to the effect from alloying elements. The effect of the final cold reduction on the tensile properties of three commercial alloys is shown in Figs. 7, 8, and 9 and includes specimens taken in both the longitudinal and transverse directions.

The difference in longitudinal and transverse properties has been a subject of considerable discussion. Some aspects of the relation of preferred orientation to this behavior have been described by Bakar-ian (7). According to Beck (13) the amount of cold rolling which can be applied in one direction is limited and varies with the differ-

ent alloys. When this limit is exceeded there is a decrease in the mechanical properties in the longitudinal direction while an increase occurs in the transverse direction. This effect can be alleviated to some extent by proper application of cross rolling; but on a commercial scale it is difficult to do this with long slabs or strips of sheet.

This effect was shown by Jones and Powell (14) to be quite pronounced on cold rolling of pure magnesium and the 1.5 per cent manganese alloy, which showed increases in tensile strength up to

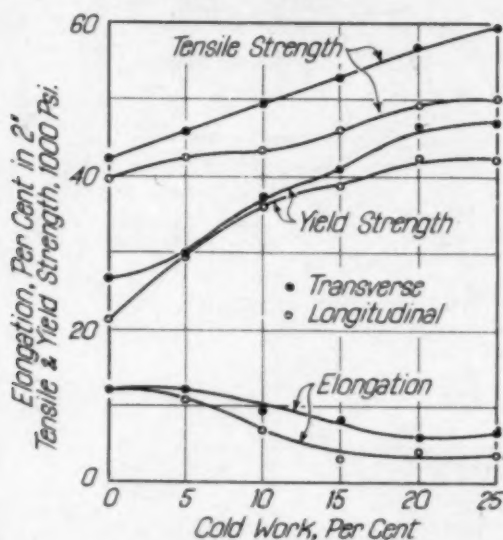


Fig. 9—Effect of Cold Work on Tensile Properties of AM-C57S.

certain amounts of cold reduction and then decreased in tensile strength with further rolling. As the cold working increased, the difference between the longitudinal and transverse properties became greater.

Beck (13) states that magnesium sheet is often rolled dry in both the hot and cold ranges because of the lack of proper lubricants and proper distribution of lubricants. Uneven distribution of lubricant causes varying rates of metal flow in local areas of the sheet, with the result that in places where the working exceeds the limit, cracking will occur locally across the direction of rolling.

These are sometimes called shear cracks and appear in cross section as shown in Fig. 10.

RESISTANCE TO CORROSION

The resistance to corrosion of magnesium alloys is greatly under-rated because of the poor behavior of the metal in the early days

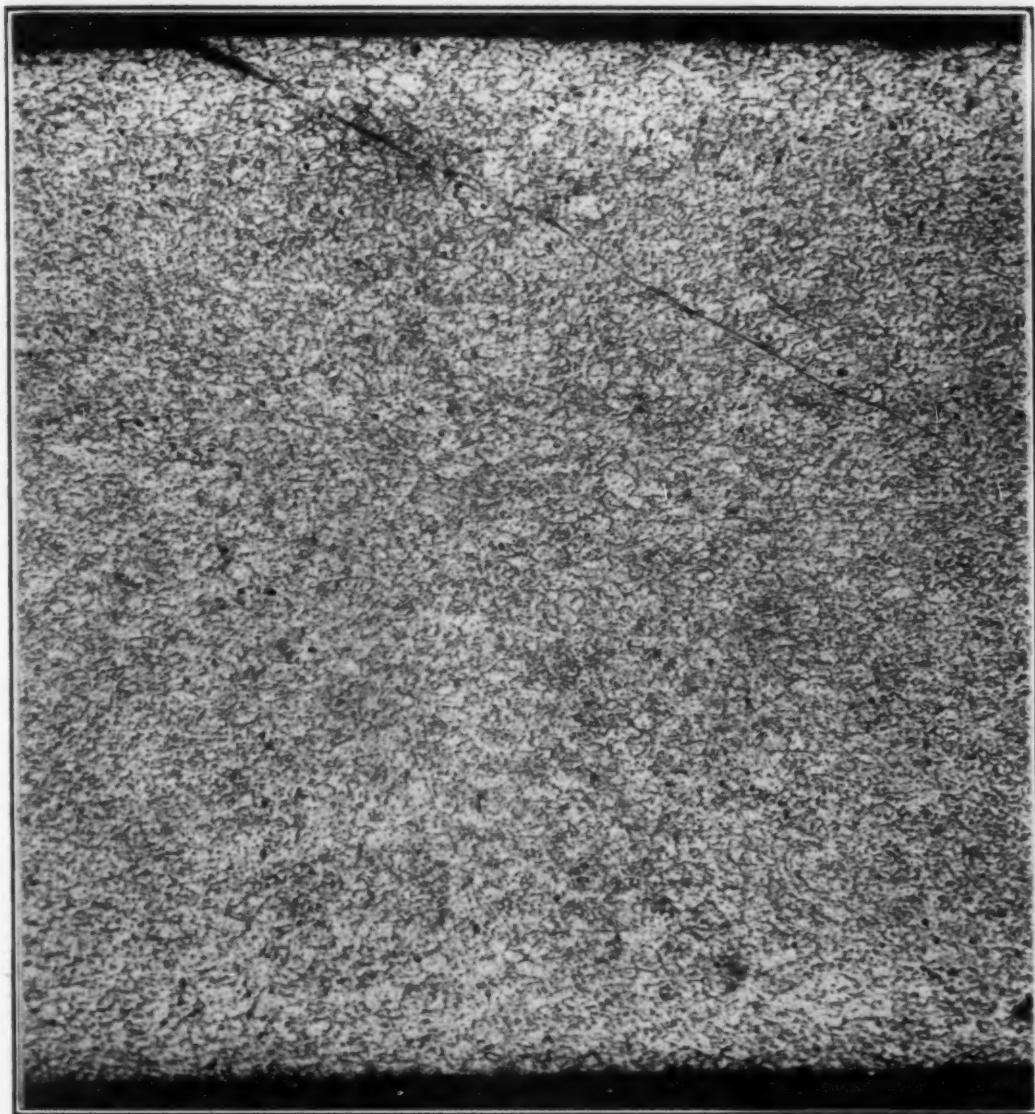


Fig. 10—Shear Crack Caused by Exceeding the Cold Rolling Limit of AM3S Sheet. ($\times 100$. Etched in Acetic Acid-Nickel Nitrate).

of its use. Since that time the inherent resistance to corrosion of magnesium alloys has been greatly improved. Magnesium of very high purity is, as a matter of fact, very resistant to corrosion and the elimination of objectionable impurities has been a major factor in improving the commercial alloys.

From the corrosion standpoint the most objectionable metallic impurities are iron, nickel, cobalt, copper and chromium; the first three being particularly bad (13), (15), (16). Hanawalt, Nelson and Peloubet (15) made a very detailed study of the effect of certain elements on the resistance of magnesium to corrosion in a 3 per

cent sodium chloride solution. Their tests were conducted on small specimens cut from slowly cooled cast samples but sheet would undoubtedly behave in a generally similar manner. In that investigation, it was shown that the corrosion rate increased very greatly when the concentration of one of these elements exceeded a "tolerance limit" for that particular element. In the absence of other elements the tolerance limits for iron, nickel, and copper were respectively 0.017, 0.0005 and 0.1 per cent. The intentional alloying additions, aluminum, zinc, and manganese, per se had no adverse

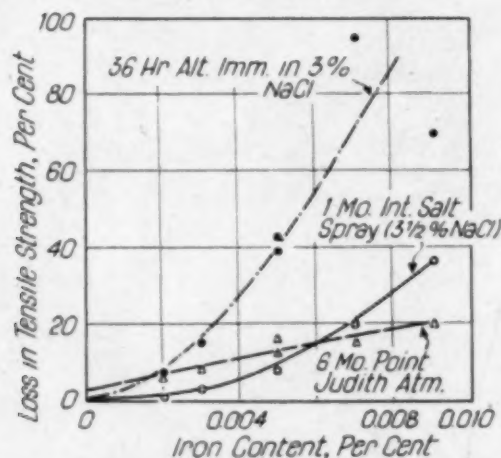


Fig. 11—Effect of Iron on the Resistance to Corrosion of 0.040 Inch AM52S-H and AM-C52S-H Sheet.

effect on resistance to corrosion, but even a small amount of aluminum lowered the tolerance limit for iron to a marked degree. Manganese on the other hand was very beneficial and substantially increased the iron tolerance of magnesium-aluminum alloys in the commercial range of aluminum contents. Zinc additions in the range employed commercially were also found to be of considerable value in overcoming the adverse effect of metallic impurities on resistance to corrosion.

When the effect of these impurities was fully realized, high purity or "C" modifications of the aluminum-containing alloys were placed on the market with limits of 0.005 per cent maximum iron, 0.005 per cent maximum nickel and 0.05 per cent maximum copper in order to provide sheet having the highest possible resistance to corrosion. These high purity modifications have now entirely replaced the corresponding alloys of lower purity.

The effect of variations in iron content on the resistance to corrosion of commercial sheet containing 3 per cent aluminum, 1 per

cent zinc and 0.2 per cent manganese (AM-C52S and AM52S) is shown in Fig. 11. The exposures included 36 hours' alternate immersion in 3 per cent sodium chloride, 4 weeks' intermittent spray using 3.5 per cent sodium chloride, and 6 months' atmospheric exposure at a sea coast location at Point Judith, Rhode Island. The losses resulting from all of these exposures increased with increasing iron contents, the rate of increase being greatest in the alternate immersion test and least for the Point Judith atmospheric exposure.

The adverse effect of impurities and the relative resistance to corrosion of the various magnesium alloys depend to a considerable extent on the environment in which the alloys are exposed, and alloys which show marked differences when exposed to sea water or in a salt solution may show no differences when exposed in a chloride-free inland atmosphere. This is illustrated by Table II which gives the losses in tensile strength for 0.064-inch sheet specimens of several commercial magnesium alloys resulting from exposure in different environments, which included salt solution, salt spray, a highly industrial inland atmosphere at New Kensington, Pa., and a severe sea coast atmosphere at Point Judith, R. I.

Table II
Effect of Environment on Corrosion Losses

Alloy	Per Cent Change in Tensile Strength				
	24 hr. Alternate Immersion NaCl-H ₂ O ₂	48 hr. 3% NaCl Alternate Immersion	2 mo. Intermittent 3½% NaCl Spray	1 yr. Pt. Judith, R. I. Atmosphere	18 mo. New Kensington, Pa. Atmosphere
AM3S-H	— 6	+ 3	— 9	—12	— 9
AM52S-H	—41	—47	—15	—22	—12
AM-C52S-H	—27	—11	— 3	—13	—12
AM-C57S-H	—22	— 1	— 8	—11	—13

The AM52S-H having a normal iron content is inferior to the AM-C52S having a low iron content and to the other alloys in the salt environments, including the Point Judith exposure, but in the industrial atmosphere at New Kensington it compares favorably with the other alloys. AM52S-H sheet is now obsolete and has been replaced by the low iron alloy, AM-C52S-H which is much more resistant to corrosion.

Stress Corrosion Cracking—Failure of magnesium sheet by stress corrosion was reported in 1937 by Siebel (17) for Elektron AZM (6 per cent aluminum, 1 per cent zinc, 0.2 per cent manganese) and Elektron A8 (8 per cent aluminum, 0.2 per cent manganese) alloys, while no failures occurred in Elektron AM503 (1.5 per cent manganese).

The lack of any earlier reports on stress corrosion of magnesium sheet is explained by the use of the 1.5 per cent manganese alloy to such a large extent that little experience was had with the high strength alloys until highly stressed applications were tried in aircraft construction.

The subject of stress corrosion cracking of metals has been comprehensively reviewed by Dix (18) who points out that such phenomena have been observed in almost all metal systems. They are referred to by different names in connection with the various metals; for example, we have the "caustic embrittlement" of steel boiler plate, the "season cracking" of brass, the "embrittlement" of lead cable sheath and the "spontaneous cracking" of the old impure zinc-base die casting alloys. Certain aluminum alloys and some types of stainless steel are also susceptible to stress corrosion cracking under certain conditions. Stress corrosion cracking is more likely to result from residual forming or assembly stresses than from design stresses or those that result from external loading, because the residual stresses are likely to be considerably higher than the others.

Since the beginning of the war aircraft manufacturers have shown considerable interest in the use of magnesium sheet for aircraft structures and the susceptibility of the high strength alloys to stress corrosion cracking has been a cause of considerable concern. The occurrence in commercial operations has been confined chiefly to welded assemblies of the higher strength alloys only, and has not been encountered in the 1.5 per cent manganese alloy (AM3S). This situation has greatly stimulated interest in the phenomenon and in the development of means for preventing such failures. Both atmospheric and accelerated stress corrosion tests are being employed in these investigations. The former have the advantage of more closely simulating service conditions but they require prolonged exposures, while in tests of the latter type, results can be obtained in a matter of hours or days. The solutions ordinarily employed in accelerated corrosion tests produce so much general attack that they are likely to mask the occurrence of stress corrosion cracking. To overcome this difficulty, R. H. Brown and his co-workers at the Aluminum Research Laboratories have developed a solution containing 35 grams of sodium chloride and 20 grams of potassium chromate per liter. This solution reveals susceptibility to stress corrosion cracking without causing an objectionable amount of general attack, and the results obtained with it correlate well with those ob-

tained in atmospheric exposure. Two methods of stressing are employed in such tests. In the one, a sheet tensile specimen is stressed under constant load as a simple beam; in the other, specimens are stressed by bending them in an arc and inserting the ends in suitably insulated slots in metal plates to maintain the desired deflection. In the latter method the approximate stress is determined by the length to which the specimen is machined.

Susceptibility to stress corrosion cracking has been found to increase with the aluminum content of the sheet. In the commercial sheet alloys, AM-C57S is more susceptible than AM-C52S, while AM3S appears to be completely immune to this type of failure. In Figs. 12 and 13 are shown typical stress corrosion cracks obtained with AM-C57S-H sheet in atmospheric and accelerated exposures. A detailed study of the mechanism of stress corrosion cracking in magnesium alloys was made by Graf (19) who proposed the determination of a limiting stress, below which no stress corrosion would occur for each specific alloy.

Annealed sheet appears less susceptible to stress corrosion cracking than sheet in the hard-rolled temper and very few annealed samples have failed in the tests that have been conducted. This can probably be explained by the fact that the stresses employed are ordinarily based on the yield strength and would, therefore, be considerably lower in the case of annealed material.

Low temperature thermal treatments have been found of some value for minimizing the susceptibility to stress corrosion cracking; but such treatments do not completely eliminate the possibility of such failure, particularly if the stresses are in the neighborhood of the yield strength or higher. Stress relief treatments applied after forming, welding, or assembly operations provide the most reliable means of eliminating stress corrosion cracking caused by residual stresses from such operations. A treatment for 1 or 2 hours at 300 degrees Fahr. (150 degrees Cent.) is very effective but results in some lowering of tensile and yield strengths of the hard-rolled sheet as shown in Fig. 18 for AM-C52S-H.

It has been demonstrated by R. H. Brown and his co-workers that stress corrosion cracking of high strength magnesium alloy sheet can be prevented by making the stressed specimens cathodic in the test solution by the application of an external potential. This principle is being utilized in a duplex magnesium sheet known as magclad sheet, the commercial fabrication of which is now being

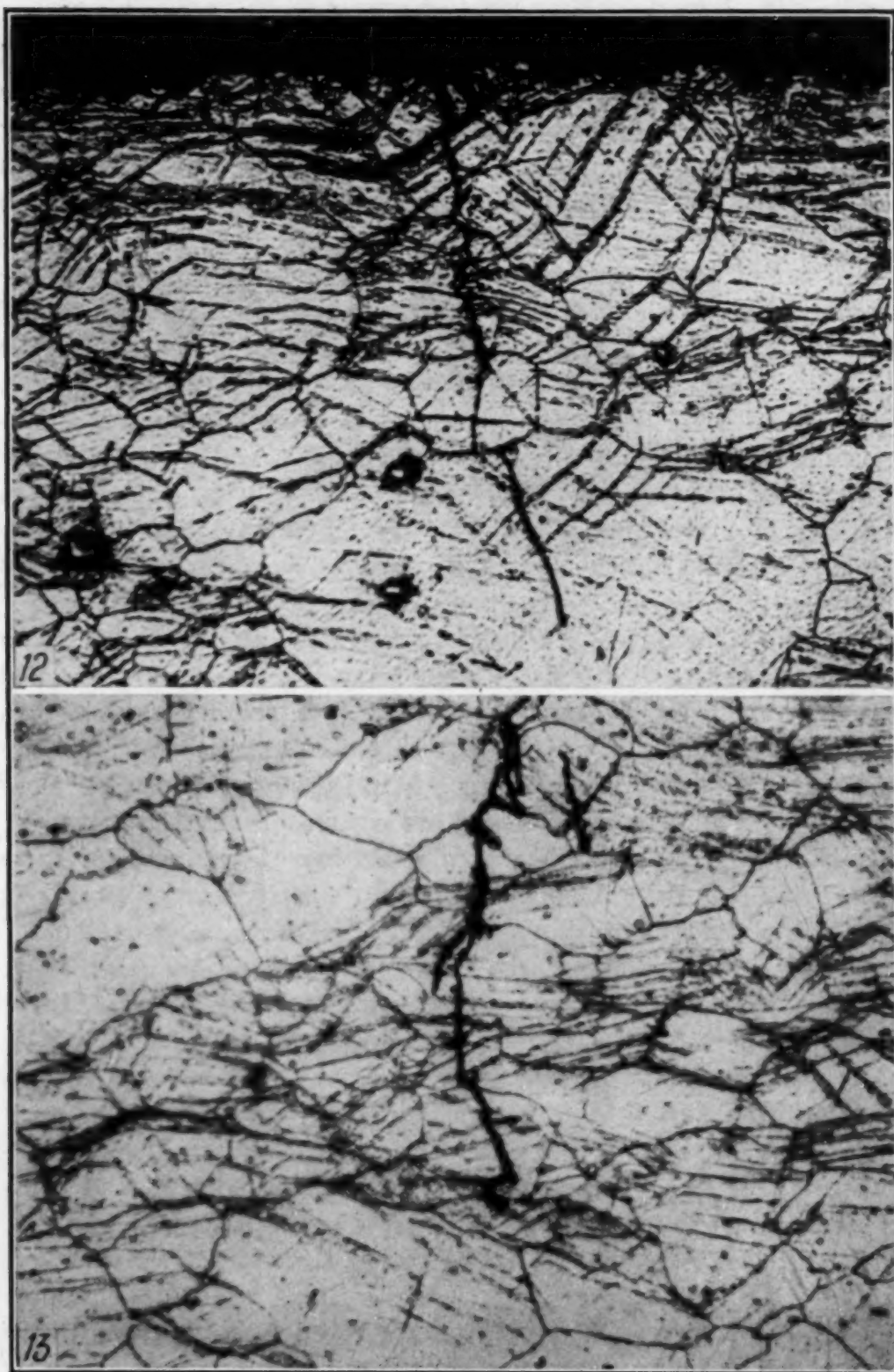


Fig. 12—Stress Crack in AM-C57S-H Sheet Exposed in Accelerated Stress Corrosion Test. ($\times 100$. Etch 50 Per Cent Acetic Acid by Vol.).

Fig. 13—Stress Crack in AM-C57S-H Sheet Exposed in Atmospheric Stress Corrosion Test ($\times 100$. Etch 50 Per Cent Acetic Acid by Vol.).

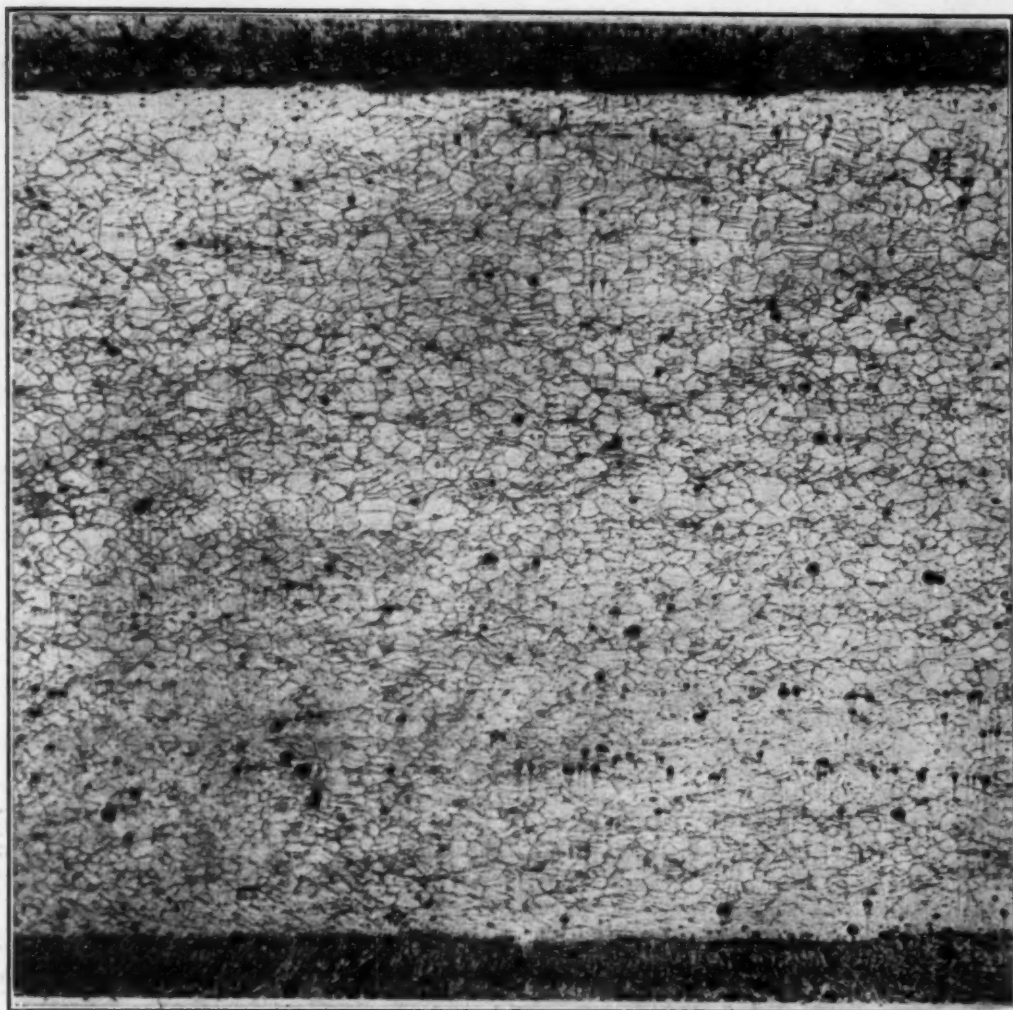


Fig. 14—Cross Section Through 0.04-Inch Magclad Sheet. ($\times 100$. Etch 15 Per Cent Acetic Acid by Vol.).

worked out. This material consists of a core of high strength magnesium alloy to which magnesium coatings having a higher solution potential than the core are bonded by special metallurgical processes. Such sheet was proposed by Brown and Willey (20) and later by Siebel. Tests conducted at the Aluminum Research Laboratories indicate that magclad sheet offers the most promising means of overcoming stress corrosion cracking in high strength magnesium alloys, and this has greatly stimulated interest in producing the material on a commercial scale. A cross section of magclad sheet is shown in Fig. 14 and a comparison of bare and magclad specimens after an exposure of 24 hours in 3 per cent sodium chloride is shown in Figs. 15 and 16. These examples illustrate not only the prevention of stress corrosion cracking in this material but also show the protection against penetration into the core by corrosion.

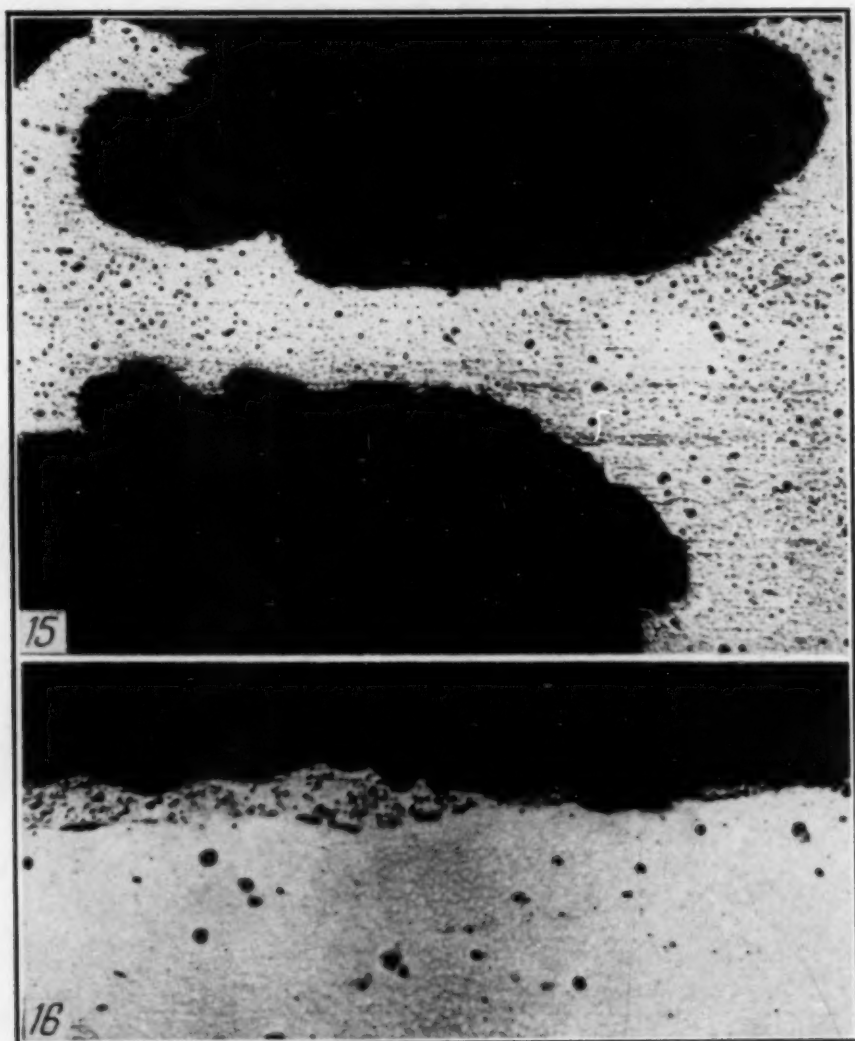


Fig. 15—Cross Section Through Corroded Area of Bare Magnesium Alloy Sheet Exposed 24 Hours in 3 Per Cent Sodium Chloride Solution. ($\times 100$. Etch 0.5 Per Cent Hydrofluoric Acid).

Fig. 16—Cross Section Through a Corroded Area in Magclad Sheet With Pure Magnesium Coating and Core Composition Corresponding to that of alloy in Fig. 15. Exposure Same as for Fig. 15. ($\times 100$. Etch 0.5 Per Cent Hydrofluoric Acid).

FORMING

The forming of magnesium sheet is easily accomplished when recognition is given to certain limiting factors which characterize the working of the material. At room temperature the forming operations are somewhat limited but the extent of forming can be increased as the temperature is raised. The principles to be kept in mind in the forming of magnesium are described by Harvey and West (21) as follows: (a) magnesium alloys must be heated to undergo severe bends, deep drawing, spinning and other relatively

difficult forming operations, (b) thin sheet heated for forming may cool rapidly during the forming operation, (c) a greater springback may be encountered when forming magnesium alloys cold than when forming aluminum alloys, (d) because of the smaller difference between the properties of annealed and cold-worked magnesium sheet and the greater rapidity of work hardening the capacity of the annealed sheet for cold forming is more limited than for aluminum, (e) the commercial magnesium sheet alloys are not susceptible to heat treatment, so when maximum properties are desired in the finished product the strongest combination of alloy and temper that will consistently withstand the forming operation is usually employed.

Cold Forming—The principal cold working operations performed on magnesium sheet are bending and drawing. The minimum cold-bend radius for magnesium sheet varies with the alloy, temper, thickness of the sheet, the type of bending equipment employed, the rate of bending, and the relation of the axis of the bend to the rolling direction of the sheet. For these reasons, an actual trial is necessary to determine the minimum bend radius that can be employed for a particular operation on a particular piece of forming equipment. As a rough guide it might be said that a bend radius of 4T can be used for 90-degree bends under shop conditions for alloys AM3S-O and AM-C52S-O and 5T for AM-C57S-O sheet ranging in thickness from 0.04 to 0.064 inch. For the hard-rolled temper these radii should at least be doubled.

The cold drawing of magnesium sheet is limited to simple shallow shapes and experience has indicated a preference for the AM-C52S composition. The aluminum content of 3 per cent in this alloy corresponds to the maximum ductility as shown in Fig. 2. The maximum reduction per draw is roughly about 20 per cent. Press equipment suitable for aluminum can be used for magnesium. The edges of the blanks should be entirely free from burrs since even an ordinary sheared edge may be the starting point for failure during cold drawing. For multiple drawing operations an intermediate anneal of 15 minutes at 650 degrees Fahr. (345 degrees Cent.) is recommended but parts may be held at this temperature for as long as 3 hours without harm.

Hot Forming—The best working temperature for the hot forming of magnesium alloy sheet is about 650 degrees Fahr. (345 degrees Cent.) for AM3S and about 600 degrees Fahr. (315 degrees Cent.) for AM-C52S and AM-C57S. Temperatures as low as 350

degrees Fahr. (175 degrees Cent.) may be used for more moderate forming operations to minimize the reduction in yield strength caused by the heating of the hard-rolled sheet at higher temperatures. At 800 degrees Fahr. (425 degrees Cent.) or higher the metal is hot short and excessive grain growth may be encountered.

The heating of annealed sheet for forming operations does not impair its tensile properties but such heating may lower the prop-

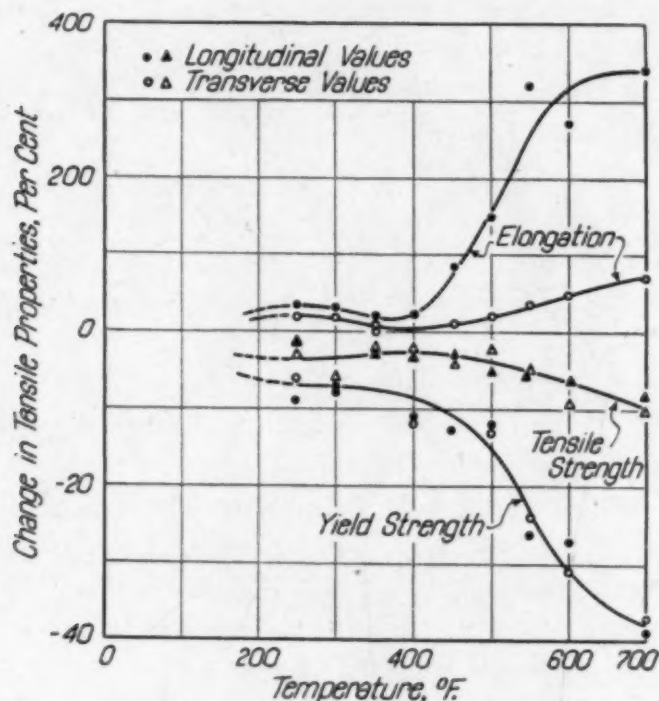


Fig. 17—Effect of Heating for 1 Hour at Various Temperatures on Tensile Properties of AM3S-H.

erties of hard-rolled sheet as shown in Figs. 17 and 18 for AM3S-H and AM-C52S-H respectively. When AM-C52S-H or AM-C57S-H sheet is heated to 500 degrees Fahr. (260 degrees Cent.), the resulting properties approach those of annealed material but the properties of AM3S-H are not affected as much although the yield strength may be reduced 10 to 15 per cent. Short time heating of hard-rolled sheet as high as 400 degrees Fahr. (205 degrees Cent.) leaves the properties of AM3S-H almost unaffected while AM-C52S-H and AM-C57S-H suffer a reduction of about 20 per cent in yield strength.

The heating of the dies to a recommended temperature of 500 degrees Fahr. (260 degrees Cent.) prior to drawing is easily accomplished by gas ring burners or electric unit heaters. The blanks

can be heated (a) in a furnace using oil, gas or electricity; (b) by a stream of hot air directed on the sheet during forming; (c) placing on a hot plate while the preceding blank is being formed; (d) immersion in an oil bath having a sufficiently high flash point. It is absolutely necessary to operate at the proper temperature to obtain uniformly good results, so active temperature control is desirable.

Hot Bending—The hot bending characteristics of the various magnesium sheet alloys are practically the same. The minimum

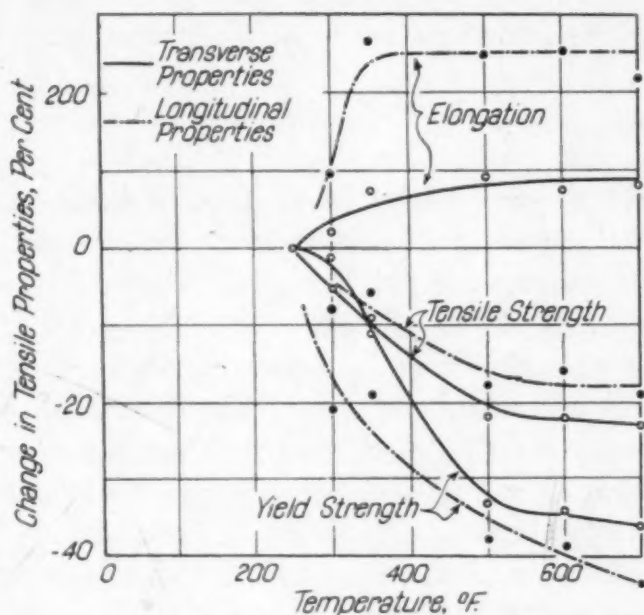


Fig. 18—Effect of Heating for 1 Hour at Various Temperatures on Tensile Properties of AM-C52S-H.

bend radius for sheet ranging in thickness from 0.04 to 0.064 inch is about twice the sheet thickness.

Hand and Hammer Work—Hand forming, with the help of wooden, cast aluminum, or magnesium forms, may be employed if the sheet parts are required in quantities too small to justify drawing tools or are of a contour which does not lend itself to drawing. Wooden, rawhide, fiber, or rubber composition mallets may be used for the actual working.

Drawing—Magnesium sheet may be successfully drawn into relatively deep shapes at temperatures in the neighborhood of 600 degrees Fahr. (315 degrees Cent.) and maximum reductions of 60 per cent have been obtained with one draw on sheet ranging in thickness from 0.04 to 0.09 inch. Maximum reductions of 50 per cent per draw are being used in production. Ordinary shop equipment

such as toggle presses, single and double acting presses are suitable for drawing operations with magnesium sheet.

Spinning—Magnesium alloy sheet can be spun if it is kept hot while being worked. Cold spinning is limited to small amounts of forming. In hot spinning the temperature is held between 450 and 600 degrees Fahr. (230 and 315 degrees Cent.) with a gas torch. There is little danger of overheating the sheet because of its rapid rotation. Recommended speeds range from 300 to 450 rounds per minute for spinning circles of 15 to 25-inch diameter, the speed depending, of course, on the severity of the operation.

JOINING

Most of the conventional methods of joining and assembling are applied to magnesium products with minor adaptations (22). Riveting and welding are the methods most commonly used for joining magnesium sheet. The newer techniques of brazing and resin bonding have shown encouraging results in experimental test. The first two joining methods are discussed in detail below.

Lock joints consisting of flanged and interlocked seams of sheet are not practical in magnesium because of the difficulty in making the necessary close bends.

Riveting—Rivets of aluminum alloy 56S are recommended for the heavier gages of magnesium sheet, for stressed construction and for the more severe exposure conditions. The electrolytic solution potential of this alloy is relatively close to those of the magnesium alloys so galvanic corrosion is minimized. For relatively thin sheet or lightly stressed parts, aluminum alloys 2S or 3S may be employed, while 53S-T and 53S-T61 are suited for joints of moderate strength. Rivets of 17S-T, A17S-T, steel or brass should be avoided because the driving pressures will damage the magnesium sheet and also because of the danger of galvanic corrosion from such dissimilar metal combinations.

Welding—An excellent and detailed description of welding practices for magnesium products has been given by G. O. Hoglund (23). Magnesium sheet may be joined by torch welding, by electrical resistance methods such as spot or seam welding and by a special arc welding procedure that employs a tungsten electrode surrounded by a shield of helium or other inert gas.

The oxyacetylene flame is preferred for torch welding and a

special flux must be employed. Filler rod having the same composition as the sheet is usually employed, but AM-C57S rod is recommended for attaching cast fittings to any sheet alloy because of the low melting point of the AM-C57S. It is essential that the welds be designed in a manner that will permit complete removal of the welding flux in order to avoid subsequent corrosion. For this reason, butt-welds are usually preferred. A cross section of a typical torch weld in AM3S-O sheet is shown in Fig. 19.

After welding, the joints should be scrubbed lightly to remove residual flux. The part is then placed in hot water 160 to 200 degrees Fahr. (70 to 95 degrees Cent.) for about 5 minutes. This removes or loosens a substantial portion of the flux which may then be flushed out. A constant flow of fresh water into the hot work tank is maintained to prevent an accumulation of dissolved flux. After this treatment the part is submerged for about 10 minutes in a 1 per cent (10 grams per liter) solution of citric acid at room temperature. This causes the formation of gas bubbles on the metal surfaces which loosen and dislodge the closely adherent flux. On removal from the citric acid solution the assembly is permitted to drain thoroughly and then is carefully rinsed in clean cold water. The parts should be dried immediately after this rinsing. This may be accomplished on a steam table or by a dip in clean boiling water.

In electric resistance welding, there is no problem of flux removal because no flux is used. The technique for welding magnesium sheet by this method is similar to that for other sheet metals except that the proper current setting must be used. A cross section through a spot weld is shown in Fig. 20. In making spot welds, it is necessary to remove the dichromate coating from the magnesium surface by means of sandpaper, emery cloth, wire brush or etching to insure consistent size of spots. This cleaning operation should be done a short time before making the welds. After welding it is necessary to again clean the spots to remove the copper picked up from contact with the electrode tips. If this copper pick-up is not removed, it will cause a galvanic corrosion of the magnesium sheet as shown in Fig. 21. Similarly both seam welding and flash butt welding can be applied to magnesium sheet and plate.

The arc welding of magnesium has recently become a practical process through the adaptation of helium shielding of the arc. This process also avoids the danger of corrosion from flux residues because no flux is required. The application of this to magnesium

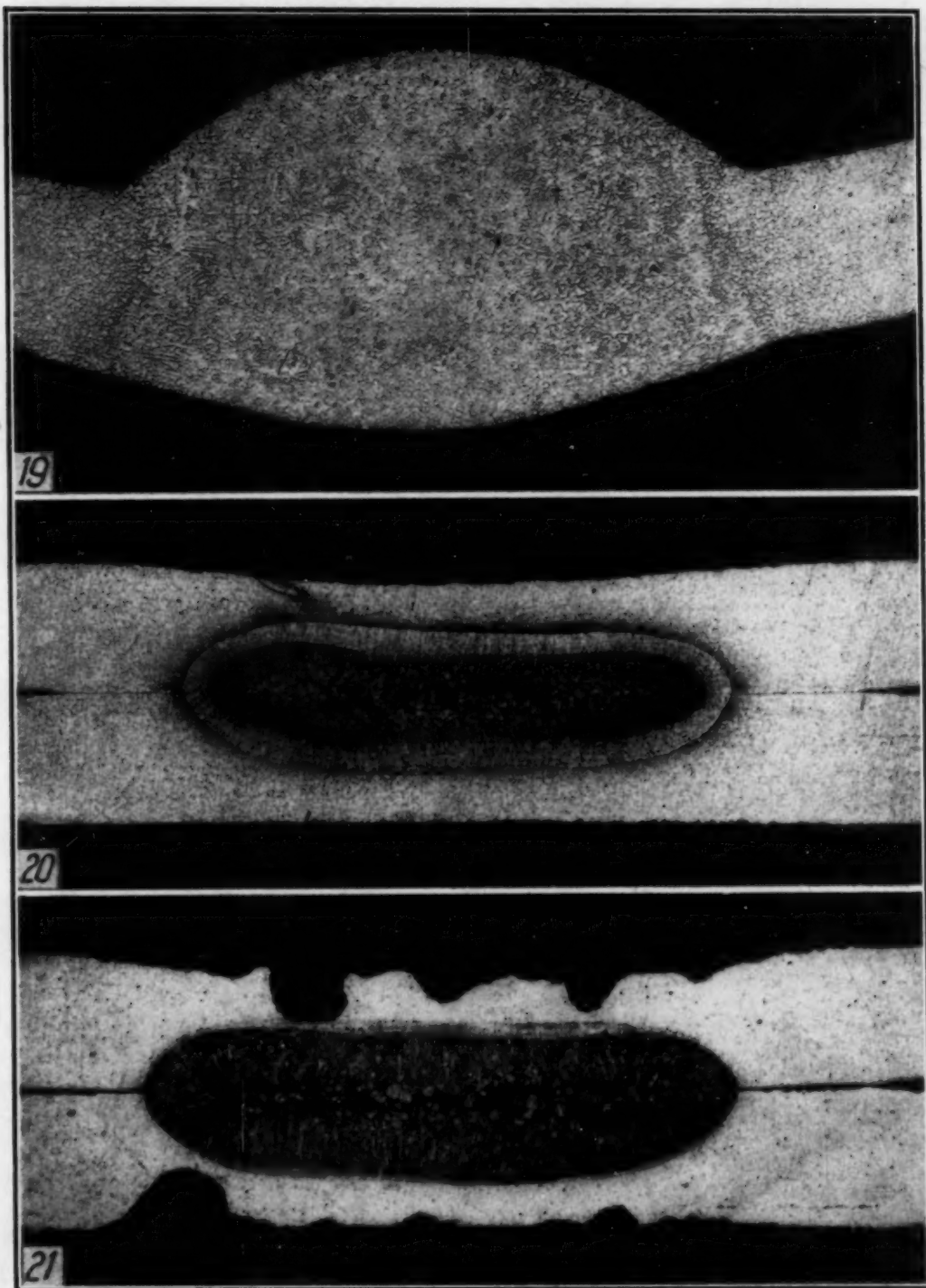


Fig. 19—Section Through Oxyacetylene Torch Weld in 0.081 AM3S-O Sheet. ($\times 10$. Etch Acetic Acid-Nickel Nitrate).

Fig. 20—Section Through Spot Weld in 0.064-Inch AM3S-O Sheet After Eight Months Exposure at Point Judith, R. I. Surfaces of Spots Were Scratch Brushed to Remove Copper Picked Up from Electrode Tips. ($\times 10$. Etch Acetic Acid-Nickel Nitrate).

Fig. 21—Same as Fig. 20 Except That Copper Pick-Up Was Not Removed Before Exposure. ($\times 10$. Etch Acetic Acid-Nickel Nitrate).

products has been described by Pavlecka (24), Piper (25), Loose and Orban (26), and Wassell (27). Because of the higher temperature of the arc, higher welding rates are possible with the helium arc than with torch welding. All of the standard sheet alloys can very readily be welded by this process. The welding rod is chosen to have the same alloy composition as the sheet being welded.

In either arc or gas welding, there is some grain growth and annealing in a zone adjacent to the weld which reduces the weld efficiency to around 70 per cent in the 1.5 per cent manganese alloy. In the alloys containing aluminum and zinc there does not appear to be any grain growth so that weld efficiencies of 90 per cent are consistently obtained.

COMMERCIAL APPLICATIONS

It has long been recognized that the light weight of magnesium alloys is a distinct advantage where portable and moving equipment is concerned. However, the early difficulties in fabrication of magnesium sheet have delayed application to such products and the poor resistance to corrosion of the earlier alloys discouraged many enterprises. The marked improvement in the resistance to corrosion in the present alloys has led to renewed development and thought in the application of magnesium.

While it is true that sheet of magnesium alloy costs more per pound than sheet of the other commercial structural metals, this is offset to some extent by its light weight which permits a greater number of articles to be manufactured per pound than from the other metals. For example four times as many objects of identical dimensions can be made from magnesium sheet as from steel sheet and $1\frac{1}{2}$ times the number from aluminum sheet. The light weight of the magnesium will also reduce handling costs in manufacturing operations and permit lower shipping costs.

In addition to light weight there are other favorable characteristics which deserve consideration for the commercial application of magnesium sheet.

- Outstanding machinability
- Excellent welding characteristics
- Good hot forming characteristics
- Nontoxic
- Nonmagnetic
- Resistant to alkalis and fluorides

On the other hand there are some inherent characteristics of magnesium sheet which are somewhat unfavorable and which must be taken into consideration. These include poor cold forming characteristics, notch sensitivity, and the necessity for protection in sea coast and marine environments.

The poor cold forming characteristics have already been discussed in the section on forming.

The notch sensitivity demands that careful attention be paid to scratches, nicks, burrs, rivet holes and edges of blanks. The deleterious effect of these notches on fatigue and impact properties of magnesium sheet appears to be quite pronounced so that it is really necessary to pay attention to this factor.

It is common practice to apply chemical coatings and paint coatings when the magnesium is to be exposed to sea coast or other marine environments. A review of the various surface treatments for magnesium alloys has been given by Schmidt, Gross and DeLong (28) while painting has been described by Wray (29) who shows that the protection afforded by the paint is increased if the sheet is made from a more corrosion resistant alloy such as AM-C57S. Mutchler (30) describes various kinds of assemblies using magnesium sheet in contact with dissimilar alloys and metals in welded and riveted construction with different coatings, which were exposed to tide water and weather in corrosion tests.

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DISCUSSION

Written Discussion: By G. Ansel, metallurgical department, The Dow Chemical Co., Midland, Mich.

The results of work on magnesium sheet at The Dow Chemical Company are in general agreement with the work presented by Messrs. Stroup, Sager and West and, therefore, we will only elaborate on a few points.

In view of the selection of sheet alloys and tempers presented in Table I, it might be well to consider their characteristics and serviceability. In Table A is presented a rating chart of magnesium sheet alloys manufactured by The Dow Chemical Company.

Table A
Characteristic Rating Chart*

Alloy and Temper	Strength	Weld-ability	Tough-ness	Cold Formability	Hot Formability	Cost
DM Ma (AM3S-O)	E	A	B	B+	A	A
DM Mh (AM3S-H)	B	A	E	E		A
DM FS-1a (AM-C52S-O)	D	D	A	A	B	B
DM FS-1h (AM-C52S-H)	A	D	C	D		B
DM J-1a (AM-C57S-O)	C	B	D	C	C	C
DM J-1h (AM-C57S-H)	A	B	F	F		C

*A = Best, highest or cheapest.

FS-1a is the sheet with good cold formability, fair hot formability, and with somewhat higher properties than Ma. This sheet has the best toughness characteristics of all the commercial magnesium sheet. Ma is used for parts requiring excellent hot formability or fair cold formability combined with good weldability. FS-1h sheet is used where highest strength combined with good toughness is required. J-1h supplements the FS-1h sheet only when the combination of strength and weldability is desired. J-1a and Mh sheet are in little demand.

On page 202, the authors discuss the decrease in longitudinal properties upon continual cold rolling while the transverse properties are increasing, as reported in the literature. We wonder if they would add some comments on their own experience. In the case of M alloy and Elektron AM537, we have found it is possible to get this lowering of longitudinal properties without harming the sheet. However, even in these alloys at times and in the case of the aluminum-containing alloys, the lowering of the longitudinal properties is caused by over cold rolling the sheet and producing shear cracks (Fig. 10) which will extend in a transverse plane and be inclined at 30 to 45 degrees Fahr. to the rolling surface. The amount and extent of cracking will influence properties and the degree to which they can be recovered upon annealing. Extensive cracking accompanied by loss in tensile strength and elongation in the longitudinal direction can be produced without harming the transverse properties; as in the latter case, the cracks are in a plane which includes the axis of the test piece. For this reason magnesium sheet should always be tested in the longitudinal direction for certification or process control.

With regard to directional properties in commercial magnesium sheet, it is our experience that when sufficient cold rolling is accomplished the transverse properties are always better than the longitudinal, both in strength and ductility in all as-cold-rolled commercial magnesium sheet. The higher the aluminum content, the lower the amount of cold roll required to make the transverse properties better than the longitudinal. Upon complete annealing, the sheet becomes relatively uniform in properties. Fig. A demonstrates the influences of cold rolling and annealing on the tensile and compression properties of J-1 alloy sheet.

The data on the effect of 1 hour at 350 degrees Fahr. (175 degrees Cent.) on cold-rolled properties are presented in view of the fact that the authors on page 207 have indicated that low temperature thermal treatments have been found of value for decreasing susceptibility to stress corrosion cracking. It has been the practice of The Dow Chemical Company to apply low temperature thermal treatments to DM FS-1h and DM J-1h.

In the section on stress corrosion, the authors state that failure of magnesium sheet by stress corrosion was reported in 1937 by Siebel (*Jahrbuch, 1937, der deutschen Luftfahrtforschung*, p. 528-531). Some further significant aspects of the stress corrosion problem are brought out by Siebel in this same reference. Quoting Siebel, "This sensitivity to stress corrosion of the high percentage wrought alloys is in contradiction to the fact that in practice stress corrosion has not occurred to the extent that perhaps one should expect, but that the high percentage alloys have proven entirely satisfactory in numerous practical tests. I might state that on the basis of preliminary static and dynamic

strength tests, sub-assemblies of two airplanes were built of Electron metal which are today still in service after six years and at quarter yearly inspections show a satisfactory condition of all parts. It is seen that by sound engineering construction and attention, the sensitivity to corrosion can be reduced to a minimum value; and we believe that cladding the magnesium alloys with high aluminum content with stress corrosion insensitive material such as AM503 will mean a step further in the development of the lightest material."

Our experience with wrought magnesium in service agrees with these observations of Siebel. We know of no record of stress cracking due to service

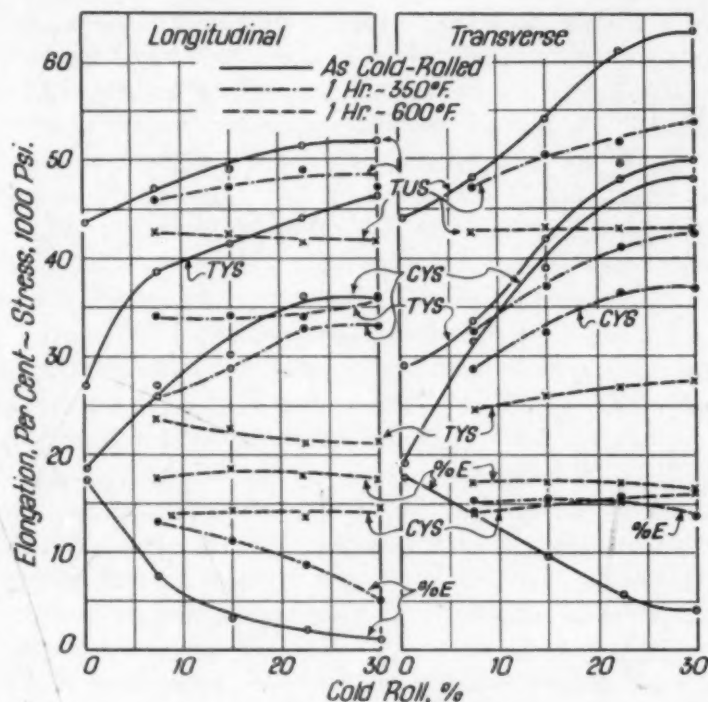


Fig. A—Effect of Cold Roll on the Properties of J-1 Alloy.

stresses. The few cases of stress corrosion cracking which are on record can be definitely attributed to faulty construction, which resulted in residual stresses in the structure. All of the evidence to date is that if the residual stresses due to welding or assembly had been eliminated by techniques now well understood, there would not have been trouble with stress corrosion cracking. Beginning in 1932 several hundred magnesium structures were built by Dow and put into service. These structures were truck and bus bodies, trailers, and canoes and were made from sheet and extrusions of magnesium with 4 and 6 per cent aluminum. With the exception of a few cases where residual stresses were known to be high, no stress cracking has been observed. Recent laboratory data on stress cracking of test bars and structures exposed at Kure Beach, N.C., have shown that present day alloys are less sensitive to stress cracking than alloys of 10 years ago. In particular, the FS-1 or AM-C52S is much less sensitive and is the recommended alloy to use for highest strength applications except where weldability is a factor. For this case J-1

or a new modification of J-1 which is equally as weldable as J-1 but is tougher and less sensitive to stress corrosion should be used. In view of the good resistance to corrosion in atmospheric exposure of these alloys and the absence of stress corrosion failures in service when properly handled, it does not appear that it is necessary to make extensive use of cladding. We fully agree with the authors that cladding will probably provide assurance of no stress cracking at constant loading at the yield stress. We have had "M" clad J-1 and FS-1 alloy sheet under test for more than 1 year at Kure Beach without any failures to date. In addition to the test bars under constant load at the yield point we have exposed welded structures of both "M" clad FS-1h and "M" clad J-1h which have shown no cracking in 9 months even though the structures were not stress relieved after welding. The sheet used for these tests was produced in the production mill in production quantities and would be available commercially if sufficient demand existed. However, we do not feel that the evidence is such as to make it necessary for a majority of applications. We would be interested in further discussion on this point by the authors as well as any comments they may care to make concerning the relative merits of cladding with pure magnesium vs. M alloy.

In considering the question of using clad magnesium base alloy sheet, one should keep in mind the high corrosion stability of alloys of the FS-1 and J-1 type in the atmosphere, even in the unprotected state. The authors have given some data on percentage strength losses on exposure under several conditions. We would like to amplify these data by giving some on exposures of bare metal at Kure Beach, N.C., 80 feet from the sea, and at Midland, Michigan. 0.064-inch J-1h sheet on the beach at N.C. without any surface protection lost 5 per cent of its tensile strength in 4 years. The same metal exposed in the industrial atmosphere at Midland lost 3 per cent of its tensile strength in 4 years. Painted samples at both locations have shown no loss in properties in 2 years.

In conclusion, the authors indicate on page 212 that heating of AM-C57S-H to 400 degrees Fahr. (205 degrees Cent.) causes a 20 per cent reduction in yield strength. This does not happen to J-1h as this temper sheet has been stabilized by a low temperature thermal treatment. J. V. Winkler, in "A Survey of Wrought Magnesium Alloy Fabrication," published on page 225 of this volume of TRANSACTIONS, indicates the small decrease in properties to be expected on heating of DM-J-1h to 400 degrees Fahr. (205 degrees Cent.). In line with low temperature annealing treatments the authors indicate that a stress relieving treatment of 1 to 2 hours at 300 degrees Fahr. (150 degrees Cent.) is sufficient to eliminate stress corrosion cracking caused by residual stresses. Our recommendations are 265 ± 10 degrees Fahr. for 1 hour for FS-1h, 400 ± 10 degrees Fahr. for 1 hour for J-1h and 500 ± 25 degrees Fahr. for 15 minutes for the annealed tempers.

Authors' Reply

G. F. SAGER: Mr. Ansel commented on the lowering of longitudinal properties that results if cold rolling is continued far enough. It has been our ex-

perience that marked decreases in longitudinal properties under such circumstances are usually associated with shear cracks. We agree that such defects may cause a substantial decrease in longitudinal properties with little or no effect on transverse properties.

We agree with Mr. Ansel that the stress corrosion cracking encountered in actual magnesium structures would not have occurred if the residual stresses introduced by forming, welding and other assembly operations had been eliminated. We also agree that the alloy AM-C52S or FS-1 is less sensitive to stress corrosion cracking than the older alloys of higher aluminum content. The elimination of residual stresses in built-up structures involves the heating of those structures, however, and it is doubtful if many of the plants making such structures would have the facilities for heating some of the largest assemblies. For this reason, we believe that the use of high strength magclad sheet not susceptible to stress corrosion cracking would be highly desirable, particularly in the case of primary structural members where a stress corrosion failure might have serious consequences.

We are pleased to note that the tests conducted by the Dow Chemical Company confirm our observations that magclad sheet is not susceptible to stress corrosion cracking. In regard to the relative merits of AM3S (Dow M) and pure magnesium coatings we can say that both types of coating have effectively prevented stress corrosion cracking in our test.

The atmospheric corrosion data given by Mr. Ansel for alloys FS-1 and J-1 exposed at Kure Beach, North Carolina, and Midland, Michigan, are of interest, and constitute additional evidence of the fact that the present day magnesium alloys are much more resistant to corrosion than most people realize.

J. B. WEST: Mr. Ansel presented a table of the various sheet alloys headed "Characteristic Rating Chart" in which were tabulated the comparative ratings for the various sheet alloys by strength, weldability, toughness, cold formability, hot formability and cost.

It is difficult to criticize the first column "strength" since such a term is so general. The following table rates the alloys by relative ultimate tensile strength. This disagrees with Mr. Ansel's comparison, principally in the relation between AM-C52S-H and AM-C57S-H. Our table also differentiates between torch and resistance weldability, no values being assigned to torch welding of AM-C57S alloy, since we do not consider its torch welding commercially feasible. A similar table on the heliarc welding of these alloys would be of value but our present lack of experience in this field prevents any reliable comparison.

We agree with Mr. Ansel's comparison of the cold formability of the sheet alloys in the soft temper but do not feel it advisable to rate the hard tempers since these are not recommended for forming or drawing in general. It has been our experience that AM-C52S-O has a slight edge over AM3S-O on severe forming which requires heat. We see very little value in Mr. Ansel's column headed "toughness" both from a usefulness standpoint and because the term has not been defined. Toughness has been defined as the capacity for energy absorption in plastic range of identical structural members stressed in bending or tension and we do not believe that this somewhat empirical value is of particular interest in a characteristic rating chart.

Rough Comparison of Magnesium Sheet Alloys

Alloy & Temper	Ultimate Tensile Strength	Weldability		Formability	
		Torch	Resistance	Cold	Hot
AM3S-O	F	A	A	B	B
AM3S-H	E	A	A	—	—
AM-C52S-O	D	C	A	A	A
AM-C52S-H	B	C	A	—	—
AM-C57S-O	C	—	A	C	C
AM-C57S-H	A	—	A	—	—

Sheet products requiring both forming and torch welding are normally built of AM3S-O material while those assemblies requiring little or no torch welding may be built of AM-C52S-O. Structures requiring maximum ultimate strength and assembled by mechanical means may be designed in AM-C57S-H.

A SURVEY OF WROUGHT MAGNESIUM ALLOY FABRICATION

By J. V. WINKLER

Abstract

As indicated by the title, this paper presents a discussion of latest developments for the fabrication of wrought magnesium alloys. The paper is divided into two parts as follows: (1) A detailed discussion of methods employed in a fabricating shop for shaping magnesium alloy parts, and (2) methods of joining component parts in fabricating a magnesium alloy structure.

It is suggested by the author that bulletins presenting supplementary information such as data on chemical treatments and painting, machine shop practice, fire prevention, etc., be obtained from one of the manufacturers of magnesium products.

INTRODUCTION

DURING the last few years the list of applications for fabricated wrought magnesium alloy parts has been steadily expanding. While fundamental methods used in fabricating these parts are essentially the same as those used in the fabrication of other commonly used structural materials there has been a refinement of methods making it possible to produce intricate structures of high quality at low cost. Volume increase is making possible continued improvements in shop procedure, which along with constantly improving alloys, mill methods, protective treatments, and reduced metal cost is firmly fixing magnesium alloys as an important material for use in applications where low weight is an important factor.

It is the purpose of this paper to present a summary of the latest practice in shaping magnesium alloy sheet and extrusions and to discuss briefly the various methods used in joining the material.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, J. V. Winkler, is associated with the Dow Chemical Co., Bay City Division, Bay City, Mich. Manuscript received July 29, 1944.

PART I

SHAPING SHEET AND EXTRUSIONS

Magnesium alloy sheet and extrusions are worked in much the same manner as other more commonly used metals and the same type of equipment generally used in metal working establishments is satisfactory. The main difference in the tools and technique is necessitated by the fact that many forming operations must be performed at elevated temperatures. Magnesium alloys have a close-packed hexagonal crystal form and at room temperature permissible deformation is limited. As the temperature of the metal is raised the allowable deformation increases very rapidly and at temperatures of 450 to 700 degrees Fahr. (230 to 370 degrees Cent.) the metal may be more severely worked than most other metals at room temperature.

Working metal in the heated state has several apparent disadvantages. There are definite advantages, however, which largely offset these disadvantages. The use of heat allows parts to be drawn in one operation which in other metals would require several redraws and subsequent anneals with a consequent increase in die and pressing costs. Springback is reduced as forming temperatures are increased, being negligible at the higher temperatures. The diameter of a drawn part can be controlled by temperature adjustment where dies are made from materials having a different coefficient of expansion than magnesium. This makes it possible to correct the size of parts which are slightly outside of permissible tolerance limits because of errors in die construction or because of material variations.

COMMERCIAL SHEET AND EXTRUSIONS

High strength properties are obtained in magnesium sheet by cold working it in the rolling process at the mill. When hard-rolled sheet (designated by the letter h) is used in highly stressed parts, the parts must be designed to permit forming at temperatures which do not result in excessive annealing.

Parts which are not highly stressed or parts requiring severe deformation are made in annealed sheet (designated by the letter a) which may be heated to high temperatures without affecting inherent metal properties.

The suffix -1 on the Dowmetal alloy designation denotes con-

trolled purity material in conformance with government specifications.

The following sheet alloys are available in this country at the present time:

Dowmetal M sheet, 1.5 per cent manganese (A.A.F. Spec. 11339), is used in the annealed condition for lightly stressed parts requiring maximum formability combined with good welding characteristics. Hard-rolled M sheet is used only in a few special cases.

Dowmetal FS-1 sheet, 3 per cent aluminum, 0.3 per cent manganese and 1 per cent zinc (A.A.F. Spec. 11340), is used in the annealed condition for parts requiring good formability coupled with higher strength than is obtained in Ma sheet and for parts requiring maximum impact resistance. Generally speaking this alloy has superior bendability and slightly inferior drawability qualities than Ma sheet. FS-1 sheet is not used for welded assemblies except where unrestrained welds are required due to its tendency to crack from welding stresses set up in restricted joints.

Hard-rolled FS-1 sheet is used where high strength combined with toughness is required.

Dowmetal J-1 sheet, 6.5 per cent aluminum, 0.2 per cent manganese, and 1 per cent zinc (A.A.F. Spec. 11338), is used in the hard-rolled condition where maximum strength is required. Its major advantage over FS-1h sheet is that it has good arc weldability. Annealed J-1 sheet is used in applications requiring good arc weldability and high strength where the forming is too severe to use hard-rolled sheet.

Table I lists the typical and specified minimum properties of Dowmetal sheet.

Magnesium extrusions can be obtained in a large number of shapes and in various alloys as follows:

Dowmetal M, 1.5 per cent manganese (A.A.F. Spec. 11336)

Dowmetal FS-1, 3 per cent aluminum, 0.3 per cent manganese, and 1 per cent zinc (A.A.F. Spec. 11320)

Dowmetal J-1, 6.5 per cent aluminum, 0.2 per cent manganese, and 1 per cent zinc (A.A.F. Spec. 11335)

Table I
Mechanical Properties of Dowmetal Sheet

Dowmetal Alloy	Tensile Strength ¹		Tensile Yield ¹ Strength ²		Compression ¹ Yield Strength ³		Elongation Per Cent in 2 Inches	
	Typ.	Spec. Min.	Typ.	Spec. Min.	Typ.	Spec. Min.	Typ.	Spec. Min.
FS-1a	37	32	22	..	16	..	21	12
FS-1h	43	38	33	26	26	20	10	4
J-1a	43	37	26	..	16	16	16	8
J-1h	47	42	34	32	27	20	9	3
Ma	33	28	15	..	12	..	17	12
Mh	37	32	29	22	20	..	8	4

¹In 1000 pounds per square inch.

²Yield strength is the stress at which the stress-strain curve deviates 0.2 per cent from the modulus line.

³Dow Chemical Co. specifications.

Dowmetal O-1*, 8.5 per cent aluminum, 0.2 per cent manganese, and 0.5 per cent zinc (no A.A.F. Spec.)

Dowmetal O-1HTA—same composition as O-1 but specially processed and aged to obtain high strength properties.

The strength of the above extrusions increases in the order listed. Dowmetal M is the cheapest of the alloys. FS-1 and J-1 are the most formable at room temperature. All of the extruded alloys are weldable.

The typical and specified minimum properties for extruded Dowmetal shapes are given in Table II.

Table II
Mechanical Properties of Extruded Shapes

Dowmetal Alloy	Tensile Strength ¹		Tensile Yield ¹ Strength ²		Compression ¹ Yield Strength ³		Elongation Per Cent in 2 Inches	
	Typ.	Spec. Min.	Typ.	Spec. Min.	Typ.	Spec. Min.	Typ.	Spec. Min.
M	36	32	20	17	11	..	9	3
FS-1	38	33	26	20	15	..	14	8
J-1	44	40	28	22	19	14	16	9
O-1	46	41	29	25	22	16	9	6
O-1A ³	49	44	32	27	27	22	7	4
O-1HTA ³	..	47	..	30	..	30	..	5

¹In 1000 pounds per square inch.

²Yield strength is the stress at which the stress-strain curve deviates 0.2 per cent from the modulus line.

³Dow Chemical Co. specifications.

*The letter A following the designation O-1 indicates this material in the aged condition.

CUTTING METHODS

Sheet may be cut by means of blanking dies, blade shears, routers, saws, or special types of rotary shears. The selection of the method depends on factors such as the number of parts to be run, the gage, the alloy and temper of the material, and the intricacy of shape. Magnesium extrusions are usually cut to length by means of band saws or circle saws. Routers are used in taking lengthwise cuts as, for example, in machining tapered wing stringers.

Blanking dies for magnesium are much the same as those used for other metals. Mild steel, tool steel, and Kirksite are used in making the dies as determined by the thickness of sheet and the number of pieces to be run. For maximum edge smoothness the punch and die are made of unequal hardness material, so a sheared-in fit providing minimum clearance can be obtained. For machine finished dies the clearance should preferably be held to less than 5 per cent of the metal thickness to be blanked.

In shears, the top blade should be ground with an included angle of from 45 to 60 degrees. The shear blade should be kept sharp by limiting its use to materials which will not dull its edge. Arc welded magnesium alloy sheet should not be sheared across a bead due to the danger of striking small tungsten particles which may have been imbedded in the sheet during the welding operation.

Conventional circle shears and nibblers should not be used on magnesium alloy sheet as they produce a rough, cracked edge. Special circle shears employing an inclined arrangement of shafts and cutters are available, however, which cut as cleanly as a blanking die.

Thickness limitations for shear cutting operations as listed above are set at a maximum of 0.064 for hard-rolled sheet and $\frac{1}{8}$ inch for annealed sheet. These thickness limitations may be exceeded if provisions are made to shear the metal while it is heated or if a second light shaving cut is taken to clean up the rough edge.

Routing may be accomplished satisfactorily on all type routers. The router bit should be a single or double flute type with smooth polished flutes to provide good chip removal. Both straight flute and up to 15-degree spiral flute routers have been used successfully. The straight flute type cuts slightly faster but the spiral flute type, which pulls the chips from the work, has less tendency to load up. Router bits $\frac{1}{4}$ or $\frac{5}{16}$ inch in diameter are used at speeds of 5000 to 20,000 revolutions per minute. A low viscosity mineral oil coolant is

recommended for less load up, and maximum insurance against fire hazard. Dry routing may be done with little fire hazard if router bit is kept sharp and chips are thrown free from bit.

Magnesium alloy sheet and extrusions are cut very readily with all types of saws. The pitch and set are greater than in other metal working saws when cutting plate or heavy extrusion. In cutting this material band saw raker set blades of 4 to 6-tooth pitch are recommended. In cutting sheet band saw blades of the type used for cutting steel are satisfactory. Either straight set or raker set blades having about 8 teeth per inch for fast cutting and about 14 teeth per inch where a relatively smooth edge is desired are used. Circular cut-off saws for small and medium size extrusions have a pitch of about 6 teeth per inch. Band saws should be provided with a nonsparking blade guide to eliminate the danger of sparks igniting the sawdust.

DRAWING OPERATIONS

1. Presses—In the past, hydraulic presses fitted with blank holding rams or cushions were recommended for drawing and forming work on magnesium alloy sheet. This recommendation still applies for parts where the slow uniform speeds obtained with typical hydraulic presses (about 2 to 4 feet per minute) are needed in making deep draws, forming parts such as boxes with small radius corners, making parts where the material must be severely stretched to make the shape, etc. Recent work on mechanical presses fitted with hydro-pneumatic or pneumatic cushions, however, has indicated that these presses are equal to and in some cases superior to slow hydraulic presses for certain types of jobs. Parts which tend to pucker on the punch, for example those having irregular rounding contours, have been formed on a toggle press having an average punch speed of 60 feet per minute with less trouble than on hydraulic presses with a speed of 3 feet per minute. The reason for the reduced puckering is probably that the greater speed gives less chance for the unsupported metal between the nose of the punch and the radius of the draw ring to cool and increase the puckering tendency. There is a definite advantage in using a mechanical press due to increased production rates. As an example, dies for one part which was being formed hot in a hydraulic press at the rate of 100 pieces per hour (which is the maximum rate the press cycle would allow) were changed to a mechanical press and run at a rate of 200 pieces per hour.

Single action presses not equipped with cushions may be used for hot drawing work by using springs or by clamping the holding plate with C-clamps or other mechanical means. These expedients are only recommended for temporary use, however, as springs lose their temper when exposed to the die temperature and the mechanical clamping means are slow and awkward to use. Control of clamping pressures is also inferior. As most presses may be fitted with cushions fairly easily, this course is usually taken.

2. *Drawing and Forming Dies*—The main difference in dies for working magnesium alloy sheet is that provisions must usually be made for heating and allowance must be made for difference in coefficients of expansion when materials other than magnesium alloys are used in the dies.

In parts where it is physically possible to form the material to the punch by the pull exerted on the blank by the pressure ring and draw ring, an open type die construction is used; i. e., the female die does not bottom on the punch. The only time mating dies have been used is in forming parts having re-entrant portions which could not be made by other means.

Pressure rings and draw rings are usually made from mild steel plates. There is no record of this material having worn out in service even though thousands of parts have been run on a large number of dies. In making dies from mild steel it is important that the material be stress relieved at 1200 degrees Fahr. (650 degrees Cent.) for 1 hour and air-cooled before finish machining to relieve stresses which might cause the die to warp in service. Even though stress relieved, some mild steel dies do distort slightly in service and heat resistant tool steel not hardened is being tried in an attempt to eliminate this trouble. Heat resisting Meehanite cast iron has also been tried and indications are that it will prove superior to mild steel in this respect. A final decision cannot be reached, however, until more service experience is obtained. While the cost of Meehanite is higher, the machining and polishing operations may be done in less time and in most dies this should more than offset the material cost.

Punches are made from steel, cast iron, magnesium alloys, and aluminum alloys. Magnesium alloys are used more than the other materials as they have the advantages of good machinability, they do not need correction for expansion due to heat, and, especially in large dies, their low weight facilitates set-up and removal of the punch from the press.

As the coefficient of expansion for magnesium alloys is greater than for steel and aluminum, allowance for the difference must be made when using the latter materials in die construction. In designing dies all dimensions which affect part size should be multiplied by an experimentally determined factor of 1.004 for steel and cast iron and 1.002 for cast aluminum when the dies are to be operated at 600 degrees Fahr. (315 degrees Cent.). If the dies are to be run at temperatures other than 600 degrees Fahr. the factor should be corrected proportionately.

A sketch of a typical die is shown in Fig. 1. In building up a die if the pedestals are to be of welded construction they should be rough machined and stress relieved before taking a finish machine cut to insure parallel surfaces. Danger of warpage due to welding stresses in the working parts of the die is eliminated by connecting these parts with bolts. The distance between pedestal supports or between a support and the edge of the die should not exceed four times the thickness of the draw ring plate as a general rule. The location of the supports on a draw ring pedestal is determined to a certain extent by the location of the pressure pins. The pedestal supports should be located in line with these pins as nearly as possible. The total minimum cross section in the draw ring pedestal supports may be determined by the formula:

$$A = \frac{(\text{Blank area} - \text{area of die opening})}{30}$$

The load on the punch is quite low and unless a bottoming die is used its pedestal need only be heavy enough to insure rigidity. If bottoming is required the pedestal should be designed for the maximum ram load which will be placed on the punch.

Structural shapes such as I-beams and channels are preferred to squares and rounds for pedestal supports as they distribute the loads better. Pedestal plates are usually made from 1-inch steel. The size of the draw ring and pressure ring should be made somewhat larger than the calculated blank size. The blank size can be quite accurately determined by making the area of the blank equal to the surface area of the formed part, allowing sufficient material for trim and for the material formed to the draw ring radius plus about a 1 to 2-inch holdback flange on parts which are not pushed through the draw ring. Footings should be provided where pressure pins are connected to a pressure ring as shown in Fig. 1 to provide

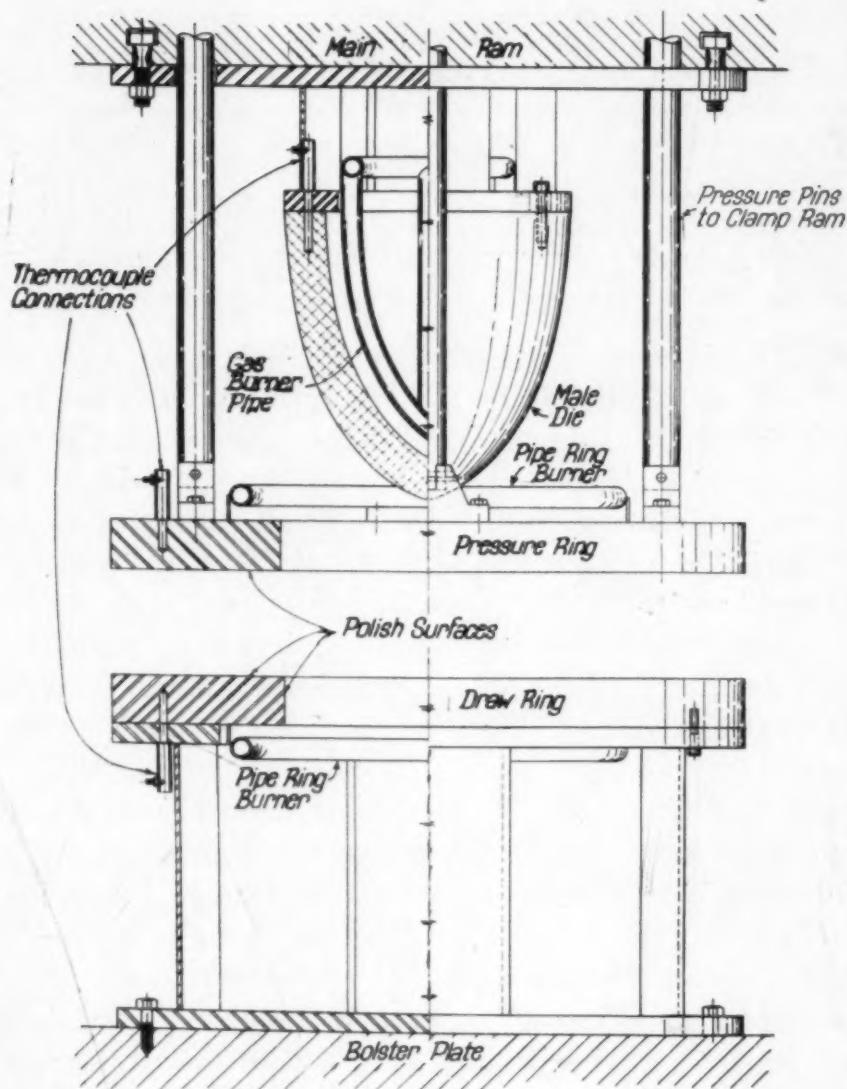


Fig. 1—Typical Die for Hot Drawing Magnesium Alloy Sheet.

maximum uniformity of pressure distribution. It is important that pressure be distributed as evenly as possible to control the tendency to wrinkle between the pressure plates and to maintain uniform material thickness in the drawn part. The minimum thicknesses of plates used for mild steel draw rings and pressure rings are listed in Table III. The thicknesses listed are for stock before machining, therefore finished thicknesses will be slightly less. Plate less than 1.5 inch thick is not used due to danger of distortion upon application of heat.

The radius of the draw ring should be made from $5t$ to $7t$ (t is the thickness of metal to be formed) for maximum drawability. Radii as small as $1t$ and a great deal larger than $7t$ have been used

Table III
Plate Thickness for Die Rings

Largest Blank Dimension Inches	Minimum Plate Thickness Inches
0-14	1.50
14-22	2
22-30	2.5
30 and over	3

successfully on draws where the flange is desired in the finished part. Radii outside the $4t$ to $7t$ limits increase drawing resistance and limit the depth of draw. Radii larger than $7t$ also increase the tendency to pucker in some parts. Box shapes should be designed with corner and bottom radii larger than $3t$ as a general rule, although flat bottomed parts have been made successfully with bottom radii as small as $1t$.

Clearance between the punch and die is a function of depth of draw. For maximum depth draws the clearance should be made equal to 0.25 to 0.35 times the metal thickness being drawn plus stock thickness.

For a limited number of parts, draw dies have been heated by playing torches over them or, in the case of small dies, by placing them in ovens. Temperatures in these cases may be checked by contact hand pyrometers or by temperature indicating lacquers, crayons, or pellets. For production work dies are heated by means of electricity or gas, and permanent temperature controlling and indicating devices installed. Electric heaters are very satisfactory but in cases where relatively high temperatures are required it is sometimes difficult to install sufficient heating elements due to the shape of the die. Both cartridge type heaters mounted in holes drilled into the die and strip heaters which clamp to the surface of the die are used, the selection being made according to the shape of the part to be heated. Temperature control is maintained by use of a controlling pyrometer which makes and breaks the heater circuit through use of a magnetic contactor.

Gas heating is accomplished by means of gas burners attached to the die in such a manner that the flames impinge on the surfaces to be heated. Gas manifolds, one suggested system being as shown in Fig. 2, are fitted to presses to be used for hot drawing work. A sufficient number of mixing and control units are provided for the maximum number of burners to be used. One burner is usually used on the

draw ring and one on the pressure ring. Whether a burner will be required in the punch or not depends on the type of draw. This will be discussed later in the paper. Burners are usually made by bending and welding 0.75 or 1-inch standard pipe in such a manner that they may

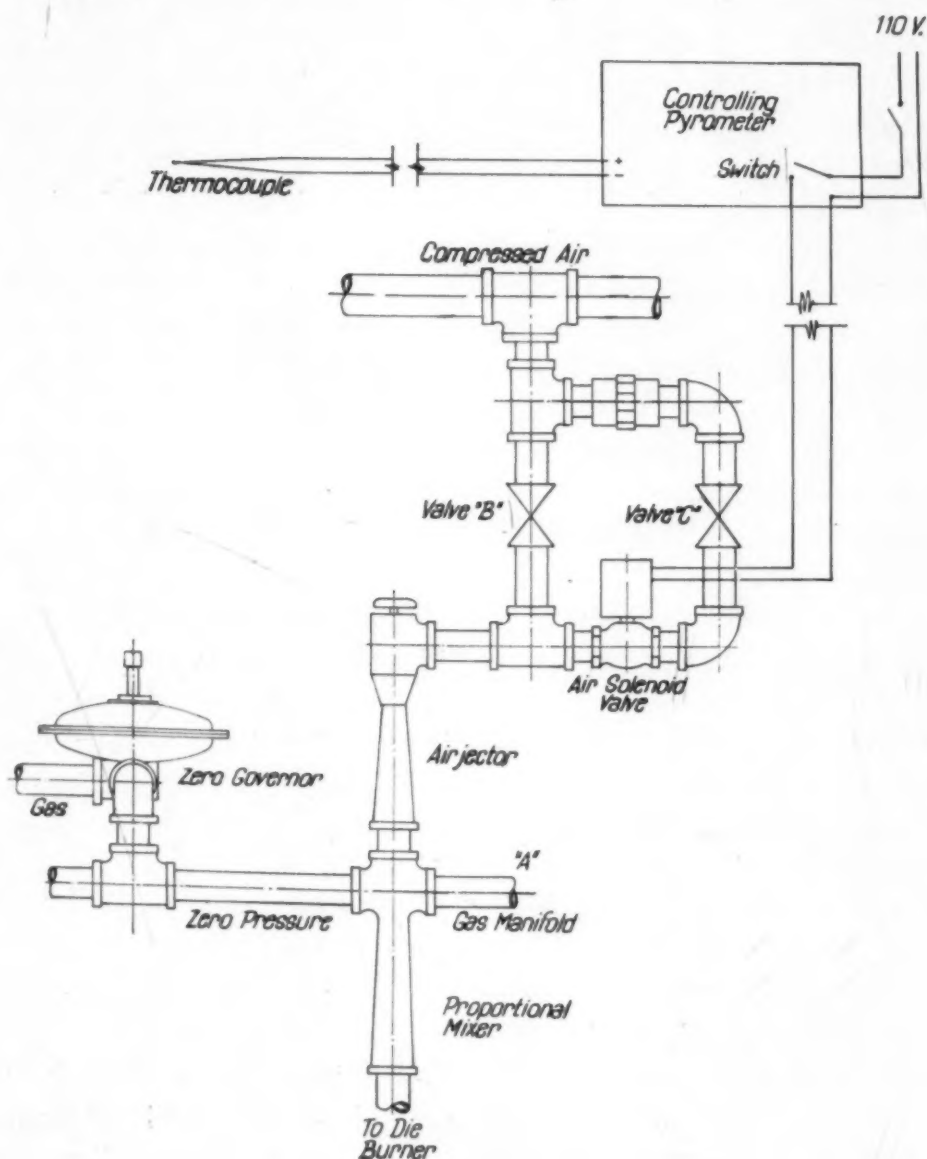


Fig. 2—Mixing Unit for Supplying the Correct Proportion of Gas and Air to a Die Burner.

be attached to the die as near as possible to the center of the draw ring or pressure ring surfaces. The burner should not be placed closer than 2 inches to the inside of a draw ring as the heat from the burner might overheat the part being formed. If the nature of the die is

such that the burner must be placed closer than 2 inches, a sheet metal shield should be used to protect the part being formed from the flame. If a burner is required in a punch it may be placed in the cavity of a hollow punch or it may impinge on the base if a solid punch is used. No. 40 drill holes spaced 0.5 inch on center are drilled in the pipe for burner ports. The pipe is so located that these ports are $\frac{5}{8}$ inch from the surface to be heated if the flame is to burn downward or horizontally, and 0.75 inch if the flame is to burn upward. Three-fourth-inch pipe is recommended for burners up to 75 inches in length and 1-inch pipe for lengths over 75 inches. 1-inch pipe has been used without trouble in burners 150 inches long. While no experience has been obtained with burners longer than 150 inches it is suspected this is near the top limit and that larger pipe would be required over this length.

The gas supply system shown in Fig. 2 operates as follows: Gas is reduced by the zero governor to atmospheric pressure in manifold A. Compressed air flowing through the airjector creates a vacuum in the proportional mixer so that gas is mixed in the air stream to form the correct proportion for burning. Valve B is set so when the die element being heated is up to temperature sufficient air flows to maintain a pilot flame on the burner. Valve C is set so that when the temperature falls and the thermocouple-controlled switch in the controlling pyrometer closes, the solenoid valve is opened and the correct amount of air will be supplied to place a full charge of mixture in the burner. With a properly set up system temperature control of ± 5 degrees Fahr. can be maintained. The cost of heating dies by this method is quite low as the average die requires only about 45 cubic feet of 1000 Btu. gas per hour during operation. When the burners are going continuously while the die is being brought up to temperature, about double this amount is used.

In locating thermocouples care should be taken to place them to give correct die temperatures. They should not be placed nearer than 2 inches to a burner unless a shield is provided to protect them from the flame.

Fig. 3 shows a typical mating die set up in a press. This photograph shows the method of mounting the burners on the die, the rubber hose used to deliver gas from the supply system to the burners and general details brought out in the preceding discussion. The use of a 0.25-inch gas line to provide an alternate means of maintaining a pilot flame on the draw ring burner is shown in the lower right-hand corner.

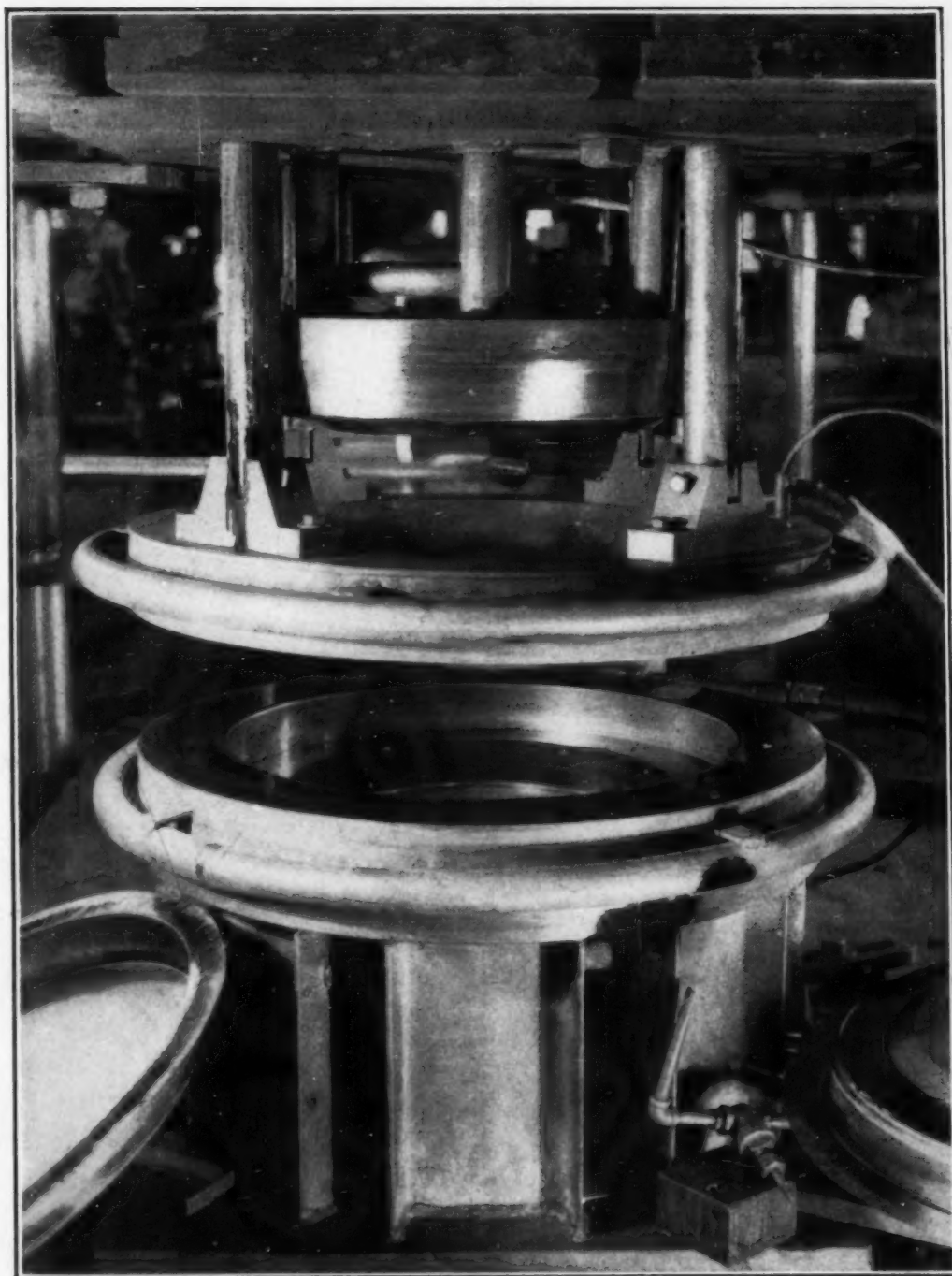


Fig. 3—Typical Mating Die for Hot Drawing and Pressing Magnesium Alloy Sheet.

3. *Preheating Ovens*—Stock to be drawn is preheated to eliminate the pause required for heating in the dies when cold sheet is used, to maintain uniform die temperatures and to gain uniform stock temperatures. In the case of round bottomed parts preheating is essential to provide heat in that area of the sheet not in contact with the

punch or draw ring and which is very susceptible to puckering if not heated to the proper temperature. While heating of flat sheet may be accomplished in atmospheric ovens a preferred method is to heat the sheet by pressing it between flat metal plates to take advantage of rapid heat transfer. One version of an oven constructed about this principle is shown in Fig. 4. Two $\frac{1}{2}$ -inch Meehanite plates with electric strip heaters clamped to them act as heaters. In the oven shown nine 1000-watt heaters are provided on each of the 30-inch square plates. This capacity will maintain a 700-degree Fahr. (370-degree Cent.) temperature on the plates under steady operation. The upper plate is held stationary and the oven is opened and closed by moving the lower plate with a pneumatic cylinder. It is important that the thermocouple used to operate the controller be located as near to the point the sheet will be heated as possible, otherwise a temperature drop due to thermal lag in the plates will be experienced. The bar shown across the lower plate drops into a recessed slot when the oven is closed. Its purpose is to lift the sheet from the plate when the oven is opened to facilitate handling. In operation one piece of sheet is placed in the oven at a time and heated as fast as an average hydraulic press can be operated. The speed of heating is such that a 0.064-inch sheet can be heated to a temperature of 600 degrees Fahr. (315 degrees Cent.) in less than 10 seconds. This makes it possible to operate a press continuously without the lost time experienced due to oven temperature drop every time a new stack of sheet is placed in an atmospheric type oven.

It is important to place preheating ovens as close to the press as possible so minimum time will be consumed in getting a heated blank into the die. This is especially true for thin sheet where the temperature drop is very rapid.

4. *Lubrication*—As many drawing operations are conducted at temperatures in the neighborhood of 600 degrees Fahr. (315 degrees Cent.) special lubricants which will not burn and which retain lubricity at this temperature have to be used. Colloidal graphite suspended in a volatile carrier has been found most satisfactory for this purpose. One mixture used quite extensively is made up of about 2 per cent colloidal graphite by weight suspended in either a low boiling point naphtha or in alcohol. This mixture is sprayed on sheet surfaces requiring lubrication by means of a standard paint spray gun in quantities sufficient to provide a uniform black surface. Application of the lubricant by means of other than a paint gun is not recommended as

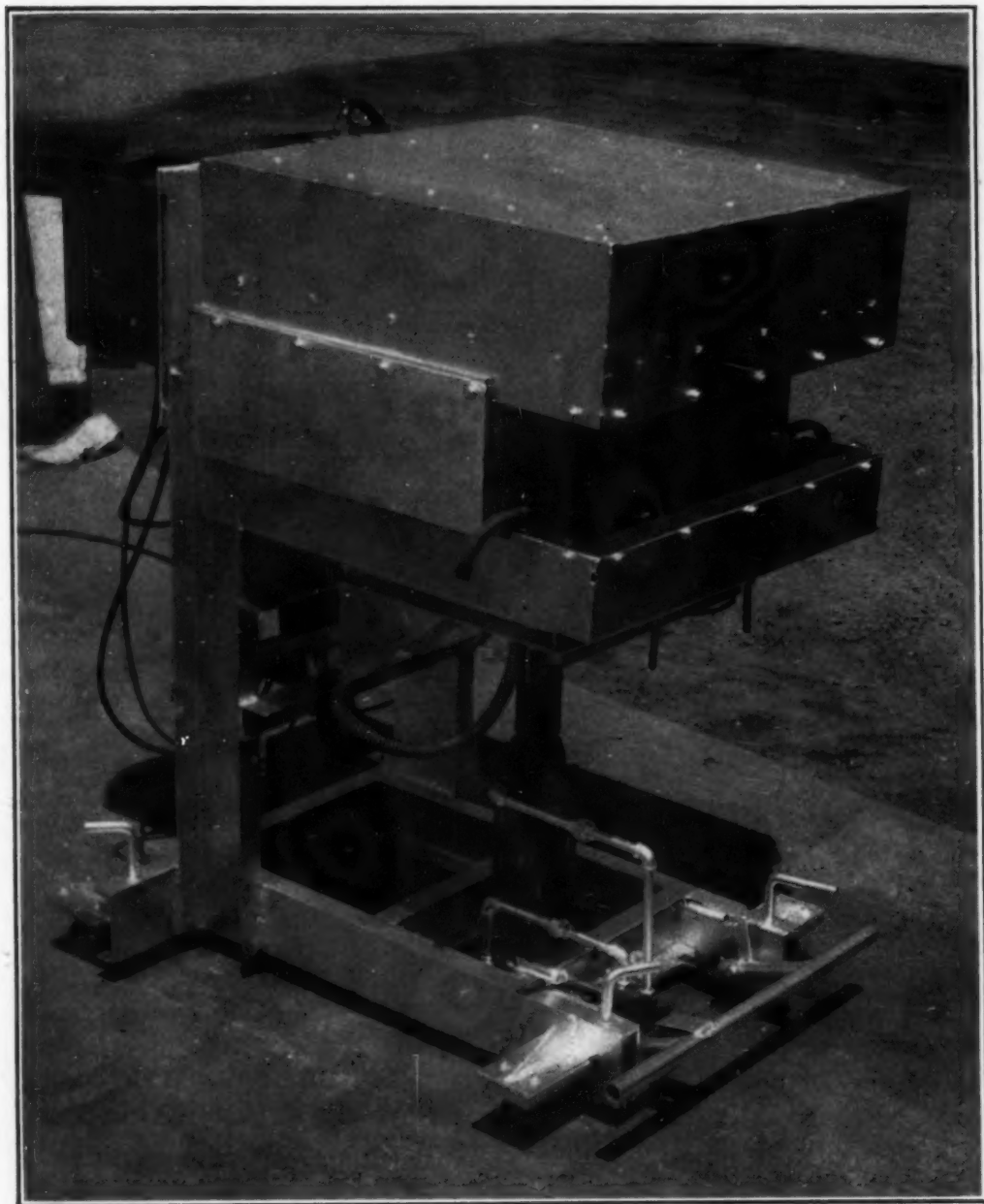


Fig. 4—Hot Plate Heater for Preheating Magnesium Alloy Sheet Prior to a Hot Forming Operation.

it is difficult to get the thickness and uniformity of coating required. The volatile carrier evaporates very rapidly and leaves a fine film of colloidal graphite which stays on during the drawing operation and provides good lubrication and freedom from scoring without fouling the die.

Die surfaces are lubricated and cleaned occasionally during operation. A graphitized grease lubricant is rubbed on working

surfaces by means of an asbestos pad. Cleaning is accomplished by rubbing with fine emery cloth.

The graphite coating on formed parts is removed as soon as possible after the forming operation by a 0.5 to 2-minute dip in a 15 to 20 per cent chromic acid bath used at room temperature. Chromic acid does not attack graphite or magnesium alloys but it does at-

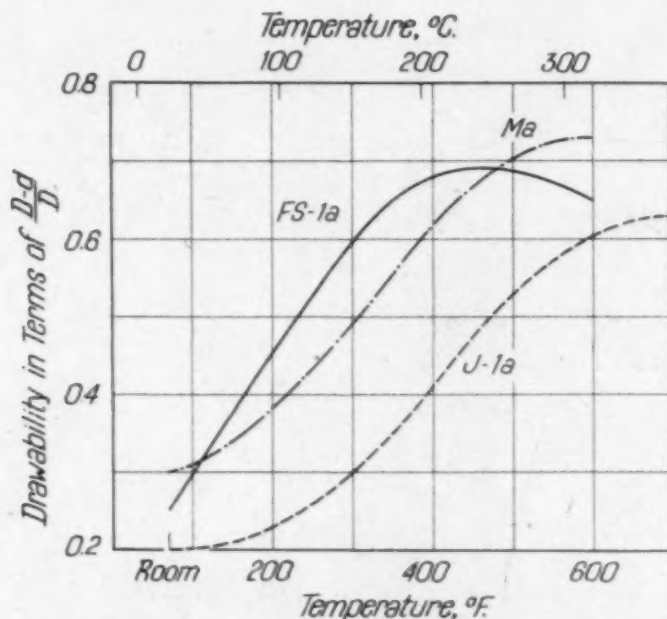


Fig. 5—Limits for Hot Drawing Magnesium Alloy Sheet in a Single Operation.

tack the magnesium hydroxide coating between the base metal and the graphite coat. This action breaks the bond between the graphite and the part. The graphite usually breaks free in the bath although on some parts which have been severely ironed a light rub with steel wool is required to effect complete removal. Parts should not be left in the bath much over the recommended time limit as the graphite:magnesium junction results in galvanic action and slight pitting will occur.

A 3 per cent addition of sodium, potassium, calcium, or magnesium nitrate appreciably improves the cleaning action of the bath. This addition is essential if the parts to be cleaned were made from pickled sheet as the absence of the magnesium hydroxide coating makes the plain chromic acid bath ineffective.

5. *Drawing Technique*—The technique required for hot drawing magnesium alloy sheet has been developed largely from the standpoint of making a part in a single operation. This was because the

extent of draw which can be obtained is so large that the great majority of parts usually can be made in this manner. Recently, however, parts with extremely deep draws have been required and double draws were necessary. The top limit for single draws is given in Fig. 5 as a function of temperature. The drawability factor $\frac{D-d}{D}$ is the ratio obtained by dividing the blank diameter minus the draw diameter by the blank diameter. As careful control is re-

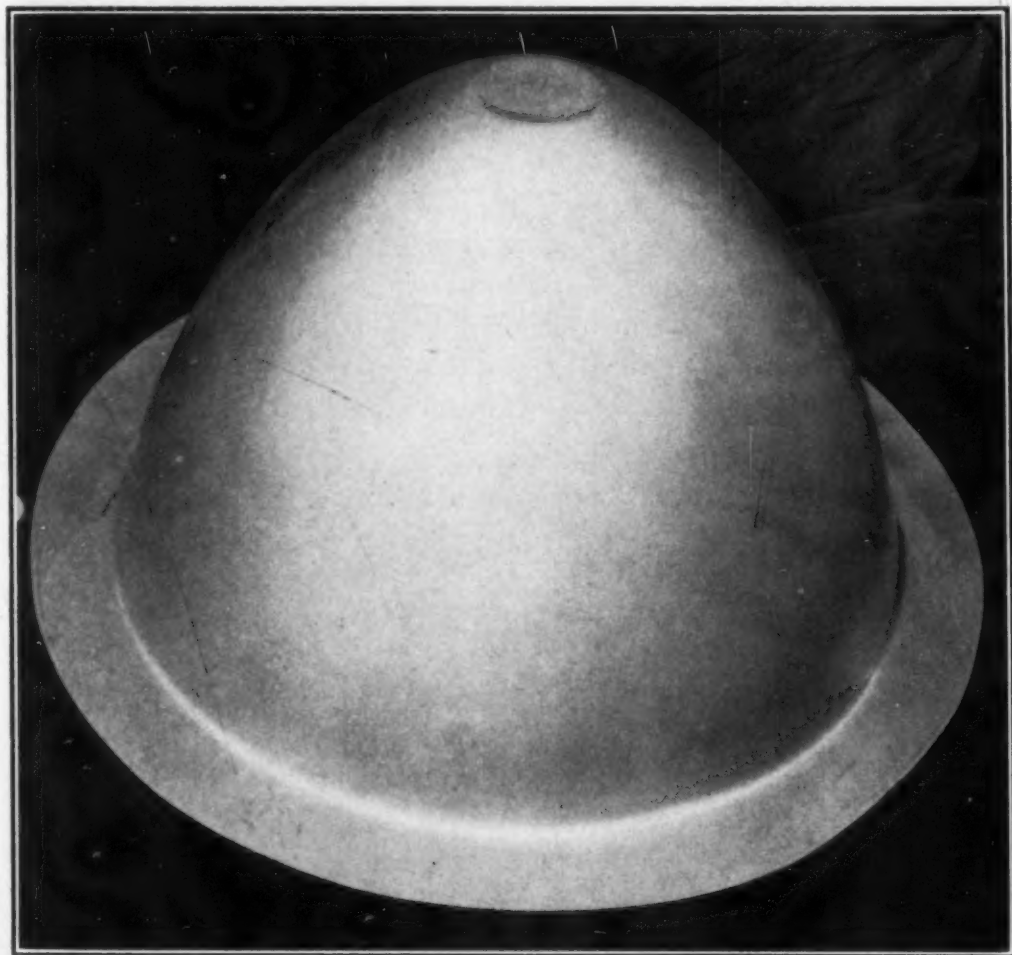


Fig. 6—Nose Spinner Drawn from 0.064 Ma Alloy Sheet.

quired to make draws of the extent shown it is recommended the top limit for a draw be set somewhat lower than the maximum given.

An example of the possibilities of using a double operation may be gained by citing a case where 24-inch blanks of both 0.25-inch FS-1a and Ma were drawn to a cup 8 inches in diameter by 16

inches deep in the first operation and redrawn to a 5.5-inch diameter cup 23 inches deep in a second operation.

The depth of draw in most parts, however, is not of primary consideration and usually no trouble is experienced in drawing to the depth required. More trouble is encountered in keeping the metal free from puckers in parts with rounding contours. In almost all

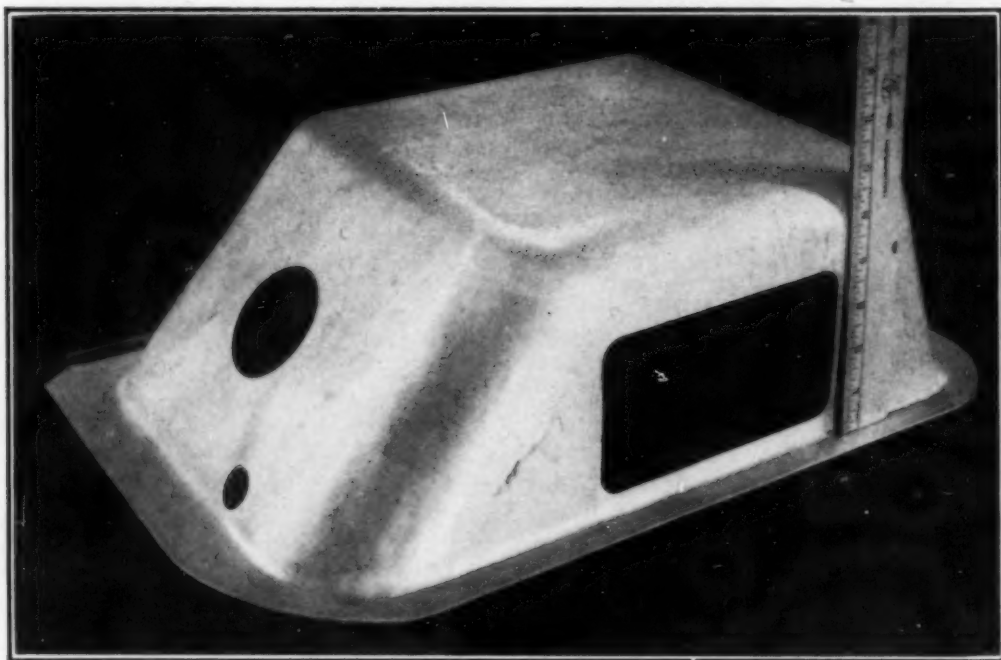


Fig. 7—Housing Drawn from 0.04 Ma Alloy Sheet.

cases, however, this trouble can be eliminated by the proper use of the tools.

Figs. 6 to 9 show a series of parts which were formed by single draws.

Fig. 6 shows a nose spinner which has been drawn in both FS-1a and Ma sheet. This part is 14.5 inches deep and has an inside diameter of 22 inches at its base. The tendency to pucker in a draw of this type is of course quite large. While the FS-1a sheet attains a maximum depth of draw at 450 degrees Fahr. (230 degrees Cent.), it was necessary to draw this part at 600 degrees Fahr. (315 degrees Cent.) (the same as used for M) to eliminate puckering. This part was drawn on a die constructed in the manner shown in Fig. 1 which was run in a hydraulic press.

Fig. 7 is a housing made from 0.40 Ma sheet. This part was made in an open type die in a hydraulic press. Considerable trouble

was encountered with puckering at the corners on the sloped end, but after the set-up was worked out correctly, scrap on quite high production runs was negligible and reworks due to small puckers less than 1 per cent.

Fig. 8 shows a power unit cover drawn from 0.072 Ma sheet and the dies used to draw it. This part is about 8 inches in diameter and 16 inches deep and was made on a hydraulic press. The colloidal

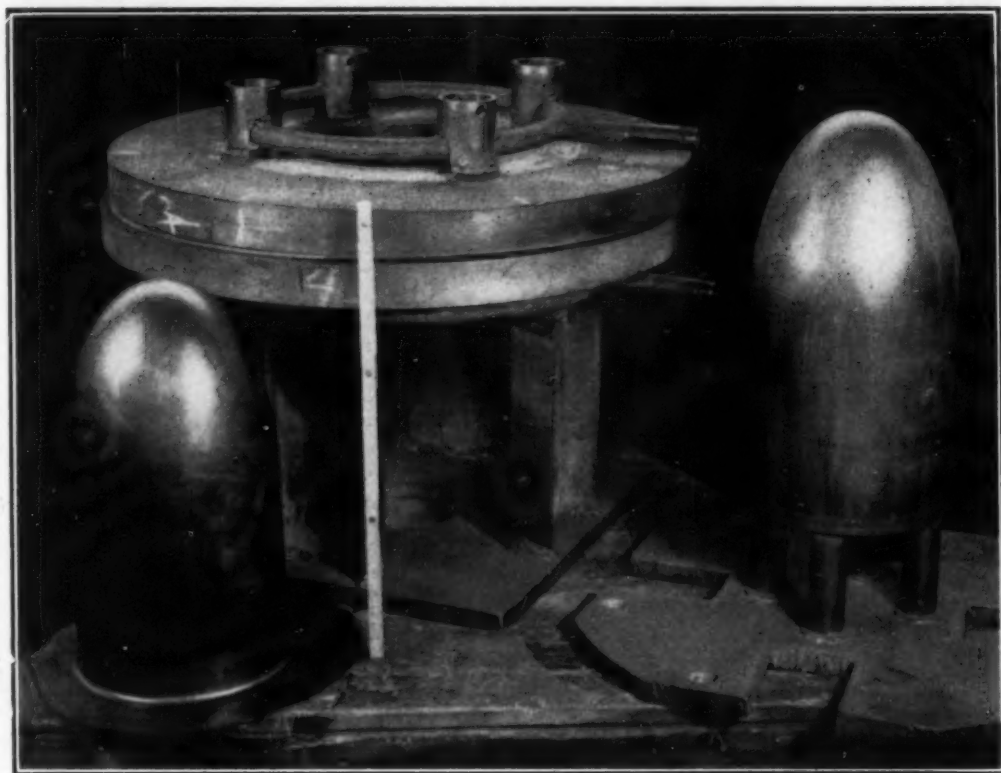


Fig. 8—As-Drawn Power Unit Cover Made from 0.072 Ma Alloy Sheet and Draw Die Used to Form It.

graphite lubricant had not been removed from the part at the time the picture was made.

Fig. 9 shows a group of boxes made on a hydraulic press. These boxes were made in both 0.040 FS-1a and Ma sheet. Box shapes such as these form very satisfactorily when the depth of the box does not exceed the length of the shorter side.

a. Temperature—The temperature required to run a given part is roughly estimated at the time the dies are designed on the basis of depth of draw and general shape. In designing dies made from material other than magnesium alloys allowance for difference in expansion coefficients is usually made for the optimum drawing tem-

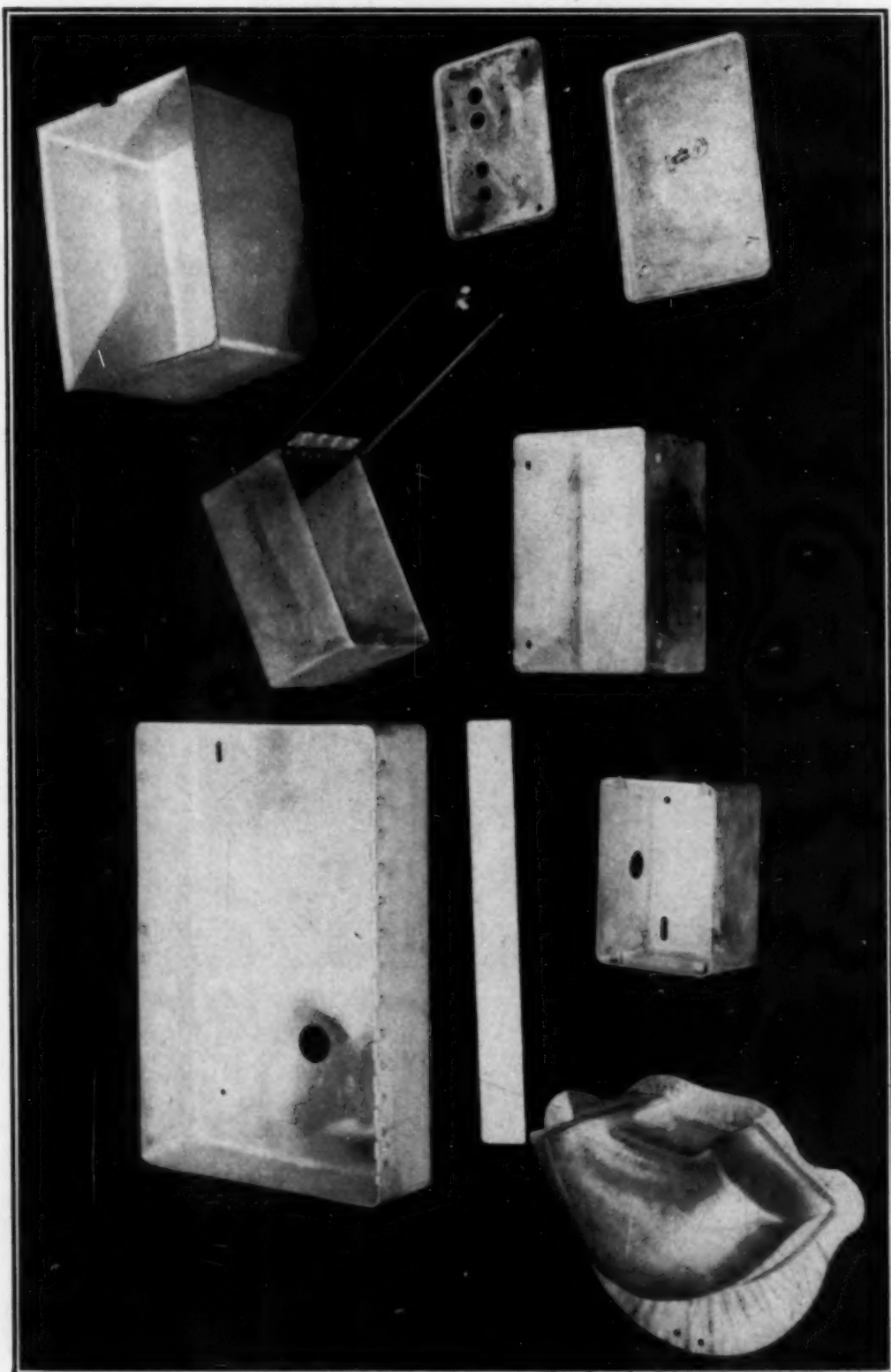


Fig. 9—Boxes Drawn from 0.040 Dowmetal FS-1a and Ma Sheet.

perature of 600 degrees Fahr. (315 degrees Cent.) for annealed sheet. Dies for forming hard-rolled sheet are designed for the maximum temperature which will not result in excessive annealing. The range of temperature which provides nearly maximum drawability is about 100 degrees Fahr. (56 degrees Cent.) above or below the temperature for which the dies were designed. In case the part is not within tolerance on its diameter, the temperature may be adjusted within this range to correct the size. For example, a part run at a temperature of 600 degrees Fahr. (315 degrees Cent.) and found to be oversize can be made smaller by running the die at a higher temperature. This results in the part being removed from the die at a higher temperature and the consequent increase in contraction more than offsets the increase in size at temperature due to expansion of the dies.

In a few cases of simple flanging operations where the metal is formed so rapidly that it does not assume die temperature, raising the die temperature will increase the part size. In cases of this type, it is possible to adjust the part size both by varying the die temperature and by changing preheat temperature.

The temperature required for the punch is determined by the shape of the part. For ordinary draws the punch is not provided with heating elements as it absorbs sufficient heat from the draw ring and pressure pad heaters. Punches for forming shapes such as hemispheres are provided with heating means so the sheet can be formed to the hot punch without puckering. For very deep draws the punch is provided with a coolant such as circulating water. The reason for cooling the punch is that while heat is provided in the blank between the holding plates making it possible to compress the sheet plastically without forming wrinkles, sufficient strength is still obtained in the sheet by cooling it as it contacts the punch to draw the sheet from between the plates. The die shown in Fig. 8 is provided with a water-cooled punch.

A unique use of temperature as a control in making parts is employed in parts having slightly crowned surfaces such as wheel fairings which must be held to quite close tolerances. Separately controlled burners are provided at the inside and outside of the die. If more crown is required, the outside of the mating die is run hotter than the inside. The part is held in the die a short time so the sheet assumes die temperature and as the outside of the sheet is hotter than the inside it contracts more after being removed from the

die and the crown is raised. For less crown the relative heat on the inside and the outside is shifted in the opposite direction.

The tensile strength and elongation of annealed sheet at elevated temperatures are shown in Fig. 10. These properties were obtained according to standard testing procedure. Forming tempera-

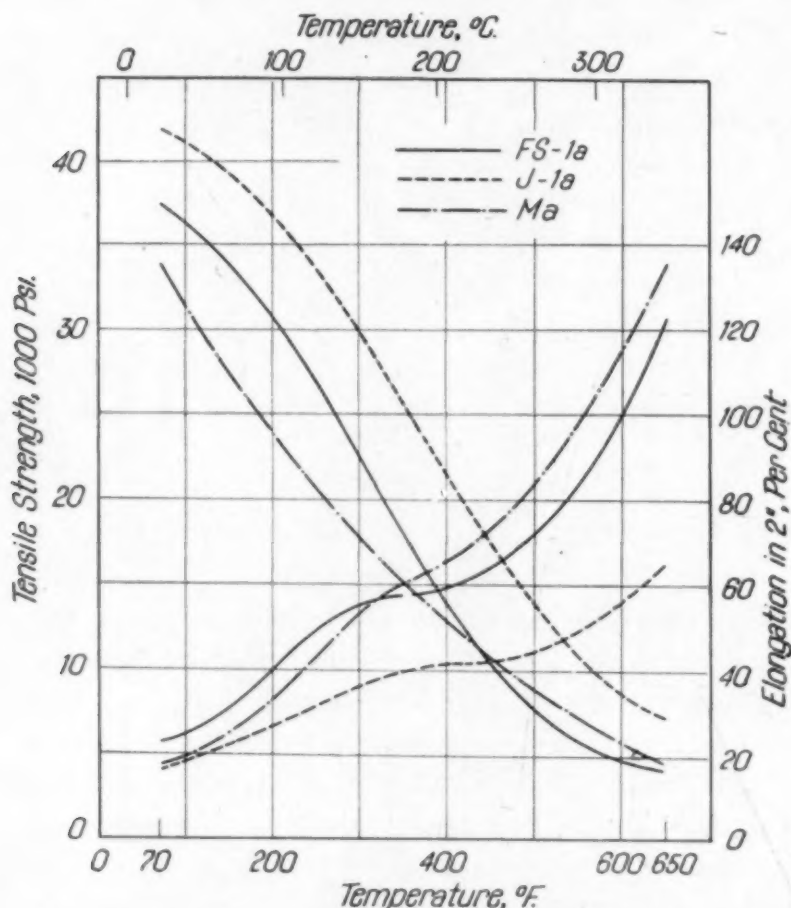


Fig. 10—Properties of Annealed Sheet at Elevated Temperatures.

tures up to 700 degrees Fahr. (370 degrees Cent.) may be used without affecting the strength of annealed sheet.

The effect of temperature on hard-rolled sheet after it has cooled to room temperature is shown in Fig. 11 for J-1h sheet and Fig. 12 for FS-1h sheet. These curves were obtained by tests on typical sheet but due to limited test data they are intended to indicate heat effect only and are not to be considered as typical or minimum values. An atmospheric oven was used in heating sheet for this test. In using these curves to indicate probable property loss in a plate type heater it should be kept in mind that a certain amount of time (about 2 to 3 minutes) was consumed in bringing the sheet up to tempera-

ture and the curves should be shifted to the left accordingly. The permissible forming temperature is limited to that which will not result in more than permissible property loss. Some operations lend themselves to rapid forming and in such cases where time as well as temperature maximums can be accurately controlled higher temperatures can be permitted.

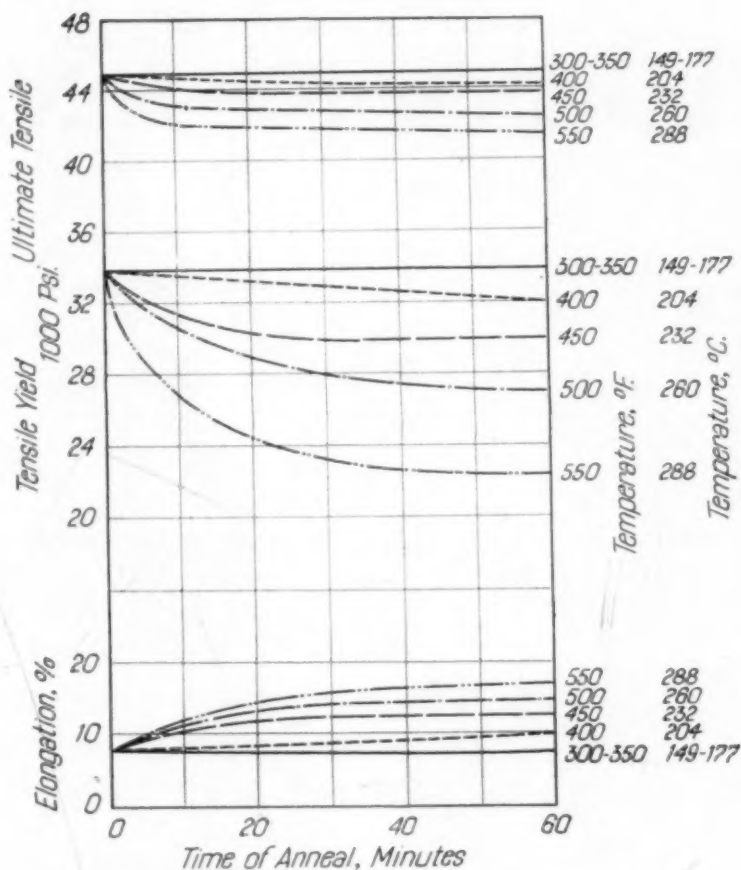


Fig. 11—Effect of Annealing Time and Temperature on the Room Temperature Properties of J-1h Sheet.

b. Holding Pressure—The blank holder pressure required to form a given part depends on a number of factors which makes an accurate estimate impossible, therefore, it is determined by trial and error. Sufficient pressure must be used to prevent wrinkle formation between the holding plates yet it must not be great enough to result in tearing in that portion which has passed the draw ring radius. For a given punch shape pressure must be increased as deeper draws are required. The depth of draw, however, is secondary in determining pressure requirements when the punch shape is such that large unsupported sheet areas exist at the start of a draw.

In drawing a hemispherical shape, for example, sufficient tension must be placed on the sheet in a radial direction to counteract circumferential compression stress set up as the punch enters the die which otherwise would cause the sheet to pucker.

Square or other irregular shapes concentrate material flow in a

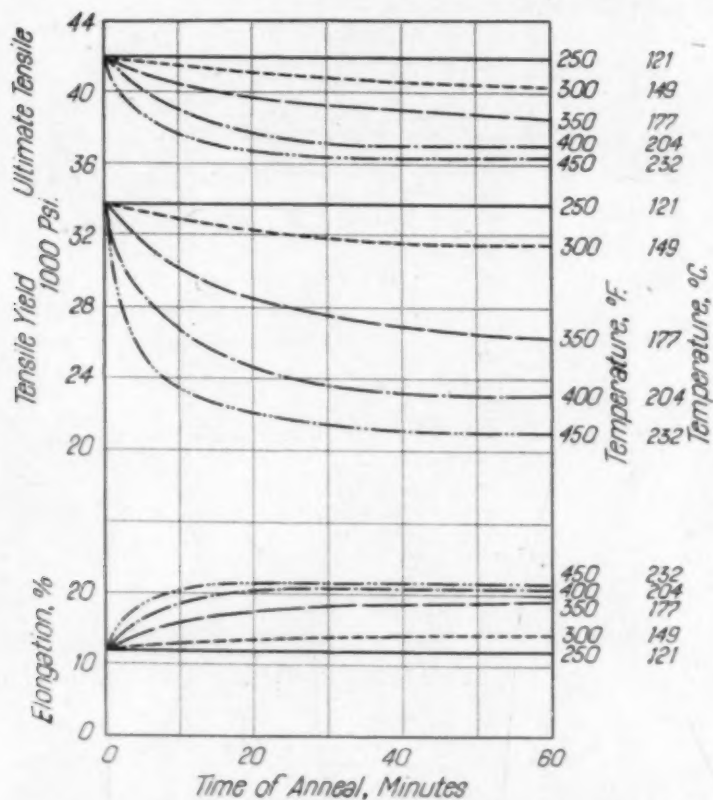


Fig. 12—Effect of Annealing Time and Temperature on the Room Temperature Properties of FS-1h Sheet.

manner which complicates holdback requirements considerably. In cases of this type draw beads, or preferably blank development, are used to obtain nonuniform restraint to compensate for unequal deformation requirements. Other factors such as draw ring radius, blank size, temperature, the type of material being formed, blank thickness, and effectiveness of lubrication further complicate the problem.

In actual practice pressures on the restrained portion of the blank have varied from the minimum which it was feasible to obtain up to 700 pounds per square inch. Most jobs, however, require pressures ranging from 50 to 200 pounds per square inch.

c. Draw Speed—The 2 to 4-foot-per-minute speed obtained with usual hydraulic presses has been found to be the most satis-

factory range for forming magnesium. The advantages of faster press speeds than 4 feet per minute have been discussed in the section on presses. The only time a slower speed than 2 feet per minute is required is when a water-cooled punch is used in making a very deep draw. In this case the speed should be less than 1 foot per minute to provide time for cooling the sheet.

RUBBER FORMING

Shallow parts such as aircraft ribs, spars and fairings which do not require drawing action are normally rubber formed (Guerin Process as developed by the Douglas Aircraft Co., Inc.). While the labor cost per piece is higher than die formed parts, the savings on die cost and the speed with which dies can be produced or revised overcome this disadvantage, especially when only a limited number of parts are required.

Special compounded natural rubbers and synthetic rubbers have been developed which may be used with dies heated up to 450 degrees Fahr. (230 degrees Cent.) very satisfactorily. Slabs of rubber, in hardnesses of about 60 Durometer, are cemented together to form a pad about 6 inches thick in the same type retaining box used for ordinary cold rubber forming. For average forming a box for a given press should be limited to a size which will enable a pressure of 1000 pounds per square inch minimum to be applied on the rubber pad.

Blocks for hot rubber forming are made from metals with melting points over 1000 degrees Fahr. (540 degrees Cent.). Metals with lower melting points than 1000 degrees Fahr. (540 degrees Cent.) which have been tried were found to creep excessively when subjected to the heat and pressure used in the operation. Magnesium alloys, either machined from plate or cast shapes, have proven the most satisfactory as no allowance has to be made for expansion differences, it heats rapidly, it is light, and is very easy to machine.

Various methods of keeping heat on the metal while it is formed may be used. The method which has been used most extensively involves the use of an oven to supply heat required. A blank is located on a forming block by means of $\frac{3}{16}$ -inch diameter or larger pins. The loaded block is brought to temperature in an oven after which it is formed in the usual manner. The forming platen should be insulated to prevent rapid chilling which would occur if the heated dies were placed on a cold steel platen.

Another method which has been used satisfactorily is shown in Fig. 13. In this set-up the platen itself is heated, and the forming block is bolted to the platen where it is heated by conduction. Blanks are heated in a preheating oven, located on the forming block and formed.

In the first method enough different parts must be available for a run so the dies may be circulated through the oven without having

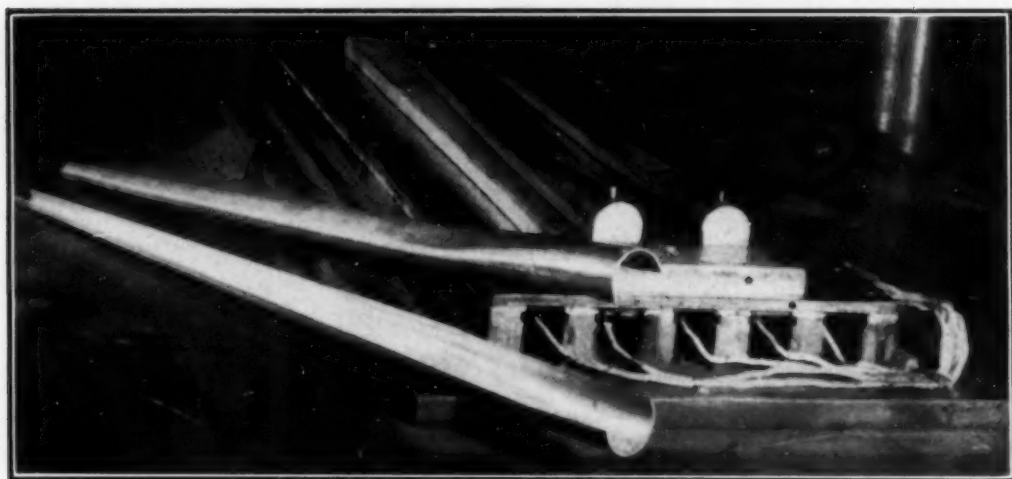


Fig. 13—Electrically Heated Platen for Hot Rubber Forming Magnesium Alloy Sheet.

to wait after a part is formed for the die and new stock to heat. This method has the advantage that if hand work is necessary on the part it may be done as soon as the part and die are removed from the press while it retains sufficient heat. The second method is usually limited to parts which may be formed directly in rubber without hand work. If rework is required in this method the part has to be reheated and formed on the die after the run has been finished and it is removed from the press. A further disadvantage of the second method is that the constant heat from the platen results in earlier breakdown of the rubber than when it is intermittently exposed to heat from the forming blocks. The second method has an advantage where hard-rolled sheet is used in that the time at temperature may be held to a minimum and higher temperatures may be used without increasing property loss as brought out in Figs. 11 and 12. If this method is used and the rubber becomes sticky the part may be protected from the rubber by covering the blank with a sheet of wrapping paper or dusting it with cornstarch just before it is formed.

Both of these methods are limited to the forming of very few parts at each stroke of the press. A method which has not been used but which should allow as large a number of parts to be formed as when aluminum parts are formed cold would be to place a number of dies on a movable heated platen. After the blanks were placed on the forming blocks the platen would be moved to an oven to insure that all blanks came up to temperature. Time in the oven

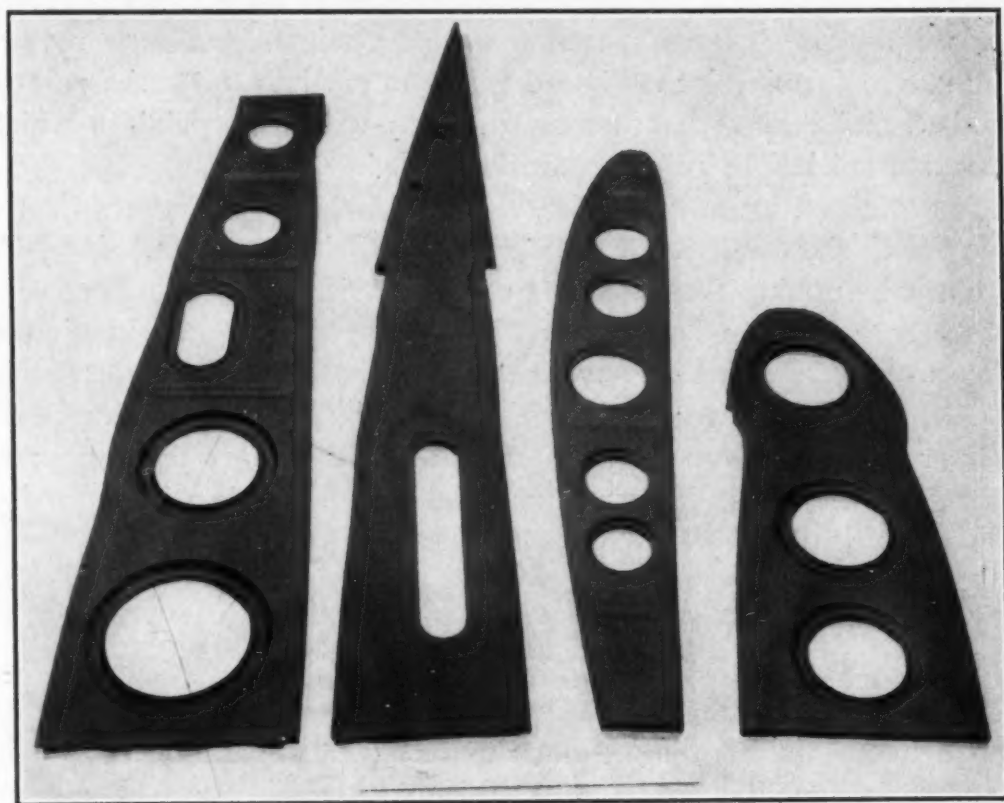


Fig. 14—Wing Ribs Hot Rubber Formed from 0.032 and 0.04 J-1h Sheet.

would be quite a bit less than in method one, probably 2 or 3 minutes would be sufficient, as the forming blocks are heated by the hot platen and they in turn would help bring the blanks up to temperature. After preheating the platen would be indexed for forming and then returned directly to the loading and unloading station. Necessary rework would be done at the press after the parts were formed as they would be held at the temperature required for rework by contact with the platen heated form blocks.

A photograph of typical rubber formed parts is shown in Fig. 14. The wing ribs shown were formed from J-1h sheet at a temperature of 400 degrees Fahr. (205 degrees Cent.). Developed

blanks with predrilled pilot holes were used so no further trim or drill operations would be required after the parts were formed until the time for their installation on a wing.

If parts are required which are difficult to make by simply pressing with the master rubber pad it often proves advantageous to use small pieces of supplementary rubber. This rubber is placed appropriately on the forming block to concentrate pressure where it is needed or to act as a spacer in starting metal to move at one location before pressure is brought to bear at other points. On parts requiring severe forming it may be necessary to partially form the part in rubber, and remove it from the press to work out wrinkles which have started before full pressure is applied.

Forming temperatures of 300 degrees Fahr. (150 degrees Cent.) for FS-1h sheet and 400 degrees Fahr. (205 degrees Cent.) for J-1h are usually used as these temperatures result in only a 2 to 3 per cent property loss when heated for 15 minutes which is the maximum time usually required. Tests have shown that at these temperatures FS-1h and J-1h have nearly equal formability. Stretch flanges of 40 per cent have been made successfully at the above temperatures. Flanges with 2.5 to 3.0 per cent shrink can be made without wrinkling but smaller radii require the use of cutouts or crimping to take care of excess metal.

HAND FORMING

The necessity of working magnesium alloys at elevated temperature makes hand forming a much more difficult operation than it is in materials which can be worked at room temperature, therefore this method is used only where a comparatively few units are required. The same technique is used in working magnesium, however, and with experience an operator can make quite difficult parts by this method.

Simple bends are made by clamping the material in a soft-jawed vise or by clamping it to a block and making the bend with a soft hammer. Form blocks may be made of a heat resistant wood such as birch although metal is usually preferred. Heating is usually accomplished by means of a torch and temperatures are checked with a hand pyrometer. In some cases where metal form blocks are used, heating is accomplished by placing the form block and sheet in an oven or by mounting electric heaters in the form block and heating the sheet by conduction.

BENDING SHEET AND EXTRUSION

a. Sheet—Straight short radius bends are made in dies by rubber forming, and by means of a press brake. Die and rubber forming methods are used in flanging class operations where bends at right angles to each other would complicate press brake operation. Various standard steel dies plus dies in which the upper blade is bottomed in rubber retained in a U-shaped bottom die are used in brake processes. Press brake dies are heated by electric strip heaters as the flat surfaces presented by this type tool are ideal for the application. Heaters are not used on a female die which contains rubber. It is recommended that dies be insulated from the press to shorten heating time, to prevent the press from heating excessively, and to conserve heat. Insulation of the bottom die may be effected easily by placing the insulating material between the press bed and bed rail. It is more difficult to insulate a top die, however; therefore a special blade holder incorporating insulation is provided for the press. High compression type insulating materials such as those used in friction blocks (which may be obtained in sheet form) should be used. The holder may be made for mounting a blade which has heaters attached to it or it may be made to incorporate the heaters as shown in Fig. 15 thereby eliminating the necessity of attaching heaters to each blade. Temperature control may be automatic or manual, aided by a contact pyrometer.

Three methods of heating magnesium alloy sheets are used. The dies may be heated and the sheet preheated, the sheet may be heated by contact with the dies, or the sheet may be preheated and the dies used cold. The latter method may be used only where the operation is fast and forming is accomplished before the dies chill the sheet. In making long bends the tendency of heat to cause warpage is reduced when the dies are used at room temperature and the sheet heated. A slight pause after the bend is made to allow the sheet to be chilled while it is held by the dies also helps in some cases. Both contact plate and atmospheric type preheating ovens are used, the latter type being essential where more than one operation is required.

Folding leaf brakes have not been used in the past because of the heating problems encountered in standard equipment. Special types, in which the bending edge may be insulated from the remainder of the machine, are being studied, however, as it is believed

Table IV
Suggested Minimum Room Temperature
Bend Radii for Sheet

Material	Minimum Die Radius	Minimum Actual Formed Radius
FS-1a	4 t	5 t
FS-1b	9 t	10 t
J-1a	8 t	9 t
J-1b	14 t	15½ t
Ma	6 t	7 t
Mh	11 t	12 t

Springback 8 to 10 degrees for annealed sheet.
 12 to 15 degrees for hard-rolled sheet.

Note: Tests made with rubber in bottom die.

the action of pulling the sheet being formed against the bending edge will eliminate the warpage problems encountered in making long free bends in a press brake.

Table IV lists suggested minimum bend radii in terms of sheet

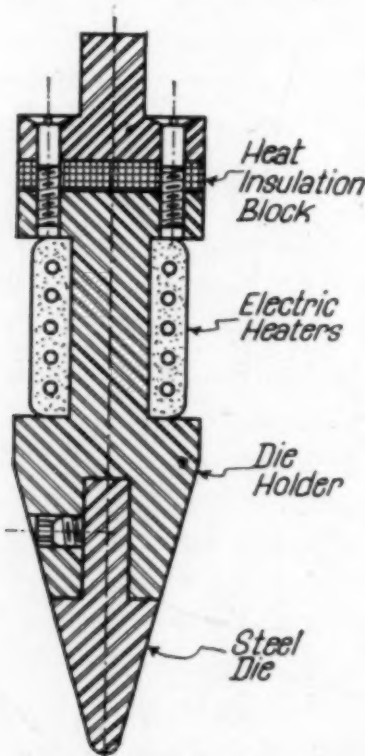


Fig. 15—Electrically Heated Press Brake Blade Holder.

thickness for room temperature forming. The data were obtained by making 90-degree bends in both directions relative to the original flat sheet surface in 6-inch wide specimens with the bend perpendicular to the grain direction. Bends parallel to the grain direction can usually

be made somewhat smaller than those listed as the sheet generally has better elongation in the transverse direction.

The curves in Fig. 16 show the effect of temperature on the smallest die radius which is normally used. These curves were obtained from testing material having typical properties but the values

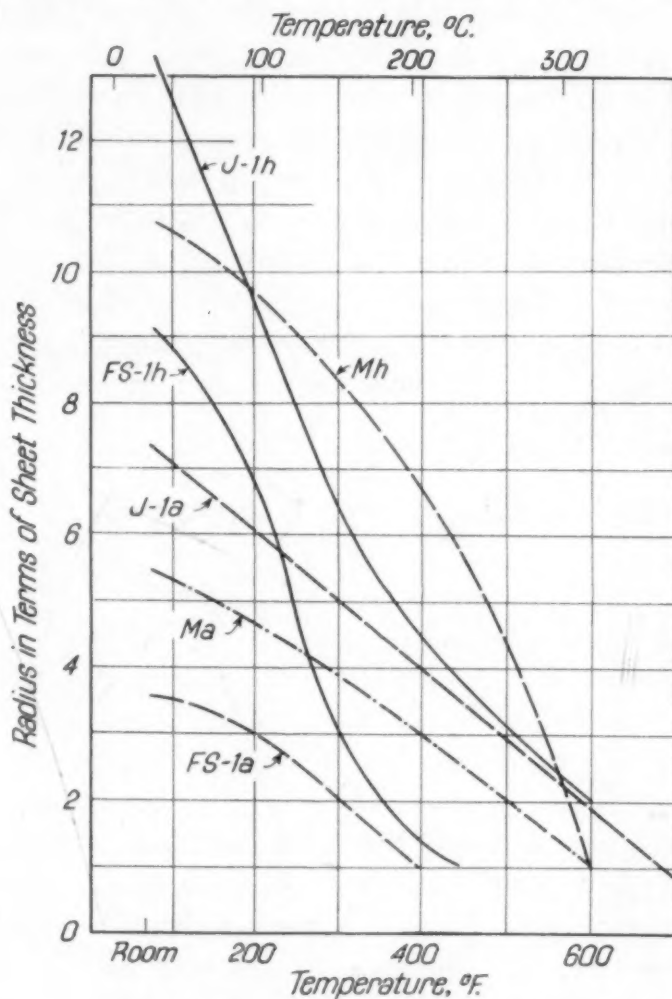


Fig. 16—Minimum Punch Radius About Which Magnesium Alloy Sheet Can Be Bent Through 90 Degrees at Various Temperatures. Test run with heated punch and rubber bottom die.

plotted are not to be considered typical due to the limited numbers of test bends made. Curves for hard-rolled sheet were extended beyond the maximum temperature usually used in forming to cover cases where fast heating is employed and maximum temperature limits consequently raised.

The above discussion relating to minimum bend radii at elevated temperatures applies when fast acting bending machines are used. By using hydraulic equipment or other machines having fairly slow

forming action smaller bends than the minimum listed can be made. Both FS-1a and Ma sheet have been formed to 90-degree bends with practically a zero radius on production items. A good illustration of the effect of slow speed on bendability is that commercial Ma sheet has been bent through 180 degrees with no radius at 600 degrees Fahr. (315 degrees Cent.) without cracking or alligating by using a press speed of 1 inch per minute to finish the bend.

Large radius bends and variable radius bends are made in sheet rolls or by passing the sheet through an operating press brake in the conventional manner. Generally the diameter of sheet rolls is larger than the minimum bend allowance for the sheet so heat is not required. In some cases, however, the sheet is heated to reduce spring-back when the roll will not form a radius as small as desired. If heat is required on press brake contouring operations both the dies and stock are heated.

Table V
Room Temperature Bend Radii for Extruded Strip

Dowmetal Alloy	Bend Radius in Terms of Strip Thickness
O-1HTA	7 to 9 t
O-1	2 to 3 t
O-1A	7 to 9 t
J-1	1.5 to 2.5 t
M	4.0 to 5.5 t
FS-1	1.5 to 2.5 t

b. Extrusions—Extrusions may be bent by hand methods, using a torch if heat is necessary and checking with a hand pyrometer. Production bending may be done on standard angle rolls, in mating dies, in stretch forming machines or other specialized bending equipment used in industry. In the relatively few cases where heating is necessary the stock may be preheated in an oven. The dies may or may not require heating depending on the size of the section and the length of time consumed in the forming operation. Experimental work with both induction heating and heating by passing current through the extrusion indicates both methods feasible in cases where high enough production is involved to warrant the expense of setting up for these methods.

Table V gives the radius about which extruded strip stock 0.090 inch thick may be bent at room temperature. While these data are not of much value in predicting the forming limits on extruded shapes, they do show the relative formability of the materials.

J-1, M, and FS-1 extrusions may be worked quite severely at 600 degrees Fahr. (315 degrees Cent.). If O-1 extrusions are to be aged after forming, forming is accomplished at room temperature as there is no advantage in heating the material up to 350 degrees Fahr. (175 degrees Cent.) which is the maximum temperature which will not result in lowered properties. Lower strength material results when the material is heated over 350 degrees Fahr. (175 degrees Cent.) due to the fact that a coarser precipitant is obtained than when it is properly aged. If O-1 extrusions are to be used in the unaged condition, forming temperatures up to 600 degrees Fahr. (315 degrees Cent.) are permissible. Forming between 350 and 500 degrees Fahr. (175 and 260 degrees Cent.) will result in partial aging with a consequent increase in strength and reduction in ductility when the material is not aged after forming. O-1A and O-1HTA material may be formed up to a temperature of 380 degrees Fahr. (195 degrees Cent.). At this temperature the bendability is about the same as that of O-1 at room temperature.

STRETCH FORMING

Only a small amount of stretch forming has been done with magnesium sheet but the work accomplished indicates this method of forming is entirely possible. Work at room temperature has been limited to annealed sheet. In working at room temperature the operation is essentially the same as working aluminum. The main precaution is that sharp, serrated grips should not be used. If a machine has grips which tend to cause failure of the sheet, or if slipping is encountered, the trouble may be overcome by lining the grips with emery cloth.

Both annealed sheet and hard-rolled sheet may be stretch formed hot. In this case the punch is made in the same manner as punches for draw dies discussed earlier in this paper.

An example of the cold stretch formability of annealed sheet is that 0.040 by 24 by 90-inch sheets of both FS-1a and Ma sheet have been formed to the contour of a 24-inch radius sphere except for 1.5 inches on the sides and the portion near the grips. A picture of a piece of 0.040 by 38 by 90-inch Ma sheet hot-stretch formed to the same contour as the above is shown in Fig. 17. It is interesting to note that the entire width of sheet formed to the desired contour except, of course, for that portion near the grips. A standard double action draw press using flat clamping plates for grips was used

in making the skin shown in Fig. 17. The punch was heated to 550 degrees Fahr. (288 degrees Cent.) and the sheet was heated by conduction as it was pulled against the punch.

DROP HAMMER

This type of forming is generally not recommended for magnesium alloys unless the part is relatively simple and can be made

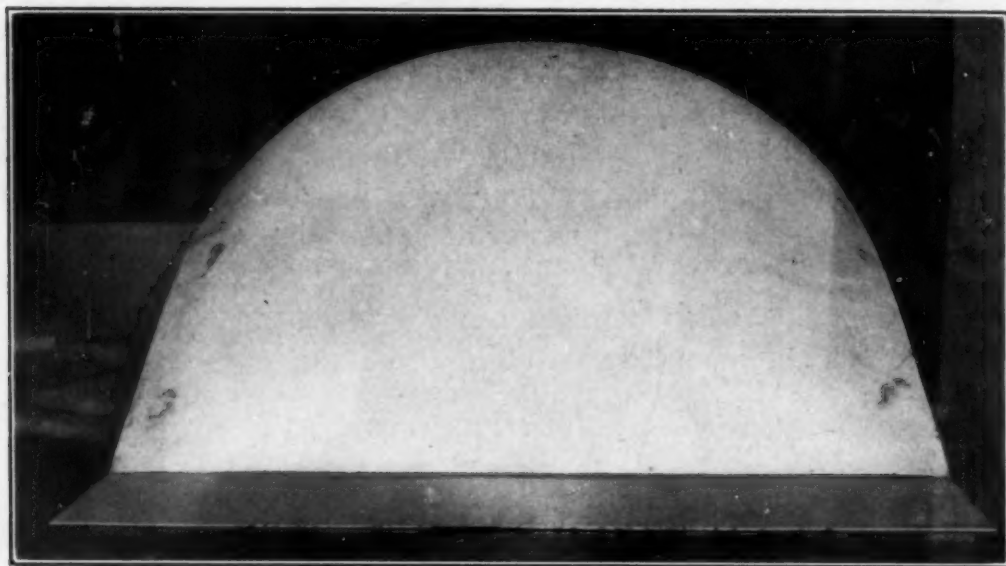


Fig. 17—Enclosure Sheet Hot-Stretch Formed from 0.04 Ma Alloy Sheet.

in one operation. The main difficulty experienced where many blows of the hammer are required is keeping the sheet heated since a partially formed sheet has only a small portion in contact with the lower die surface.

SHRINKING

Limited work on shrinking machines has proven that annealed materials may be shrunk satisfactorily at room temperature. This fact has been adequately demonstrated by shrinking one edge of a 0.040 by 1 by 9-inch sheet of Ma to an extent which resulted in the sheet bending about the neutral axis of the 1-inch width to a radius of 6 inches. Hard-rolled material cannot be shrunk very far without employing heat to increase ductility. Special grips have been developed for magnesium which do not have a stipple and consequently allow magnesium alloys to be shrunk without the objectionable surface marring often associated with this type of operation.

SPINNING

The utility of this method for working magnesium alloys in making limited quantities of parts or for accomplishing certain type operations which would be difficult or impossible by other means is

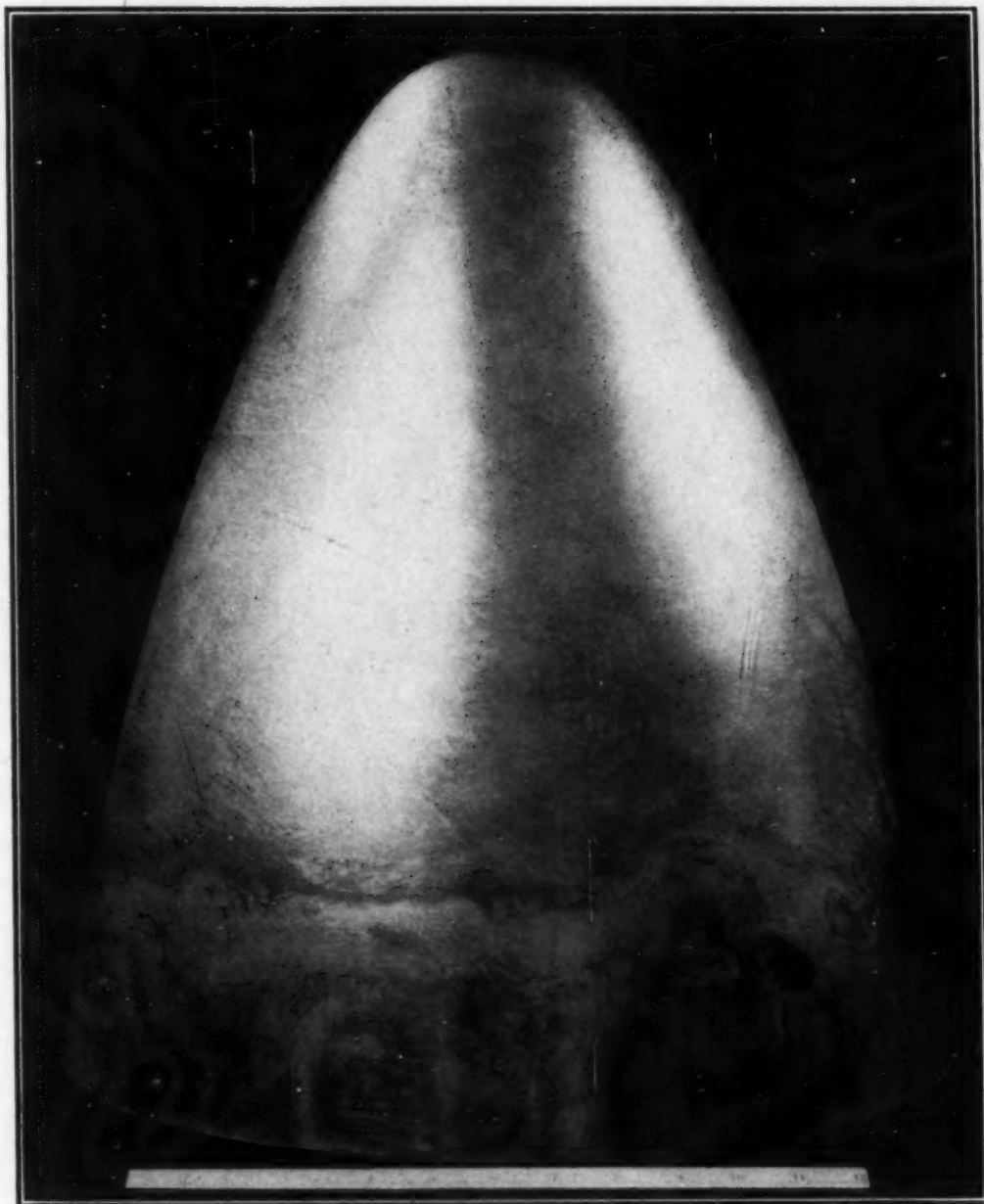


Fig. 18—Cone Made by Hot Spinning 0.051 FS-1a Sheet.

borne out by a statement made by an executive of one of the leading spinning companies in this country. He said that his company can hot spin any shape from either annealed Ma or FS-1a sheet that

can be spun from pure aluminum sheet. A photograph of a part spun from both 0.051 FS-1a and Ma sheet is shown in Fig. 18.

Heat for spinning may be applied by means of a hand torch or a heated chuck may be used and the sheet heated by conduction. Maple chucks have been used satisfactorily when a torch was used for heating but a metal chuck is used where large numbers of parts are required or where the chuck is to be heated. Standard spinning tools with brown laundry soap for lubrication have been used with good success in hot-spinning operations.

IMPACT EXTRUSION

The term impact extrusion as applied to forming shells from magnesium alloys which have too great a depth-to-diameter ratio for a drawing operation is a misnomer. While the operation is essentially the same as impact extrusion it is accomplished in a hydraulic press at much slower speeds and might more accurately be termed a back extrusion. Work with this forming method has not been extensive enough to establish related limits on wall thickness, diameter, and height. To date shells ranging in size from 1.25 inch O.D. by 6.5 inch long with a wall and bottom thickness of $\frac{3}{32}$ inch to a size 5.5 inch O.D. by 22 inches long with a wall and bottom thickness of 0.25 inch have been made from extruded M, FS-1, and pure magnesium stock.

Temperatures of 600 to 800 degrees Fahr. (315 to 427 degrees Cent.) have been used on the three materials tried to date. The pressures found necessary per square inch of punch cross section were approximately 25,000 pounds per square inch for pure magnesium, 35,000 pounds per square inch for M, and 40,000 pounds per square inch for FS-1. Ram speeds of 2 to 3 feet per minute resulting in extrusion speeds of 10 to 15 feet per minute were found satisfactory.

The main feature in die design is the necessity of reducing the cross section of the punch back of its nose to prevent the extruded metal from seizing the punch. The die used to form the 1.25-inch shell mentioned above is shown in Fig. 19.

A series of cups with various shapes forged integral with the cup base at the time of extrusion is shown in Fig. 20. The cup at the right was provided with a steel base by forging metal around a piece shaped as shown in the sectioned cup second from the right.

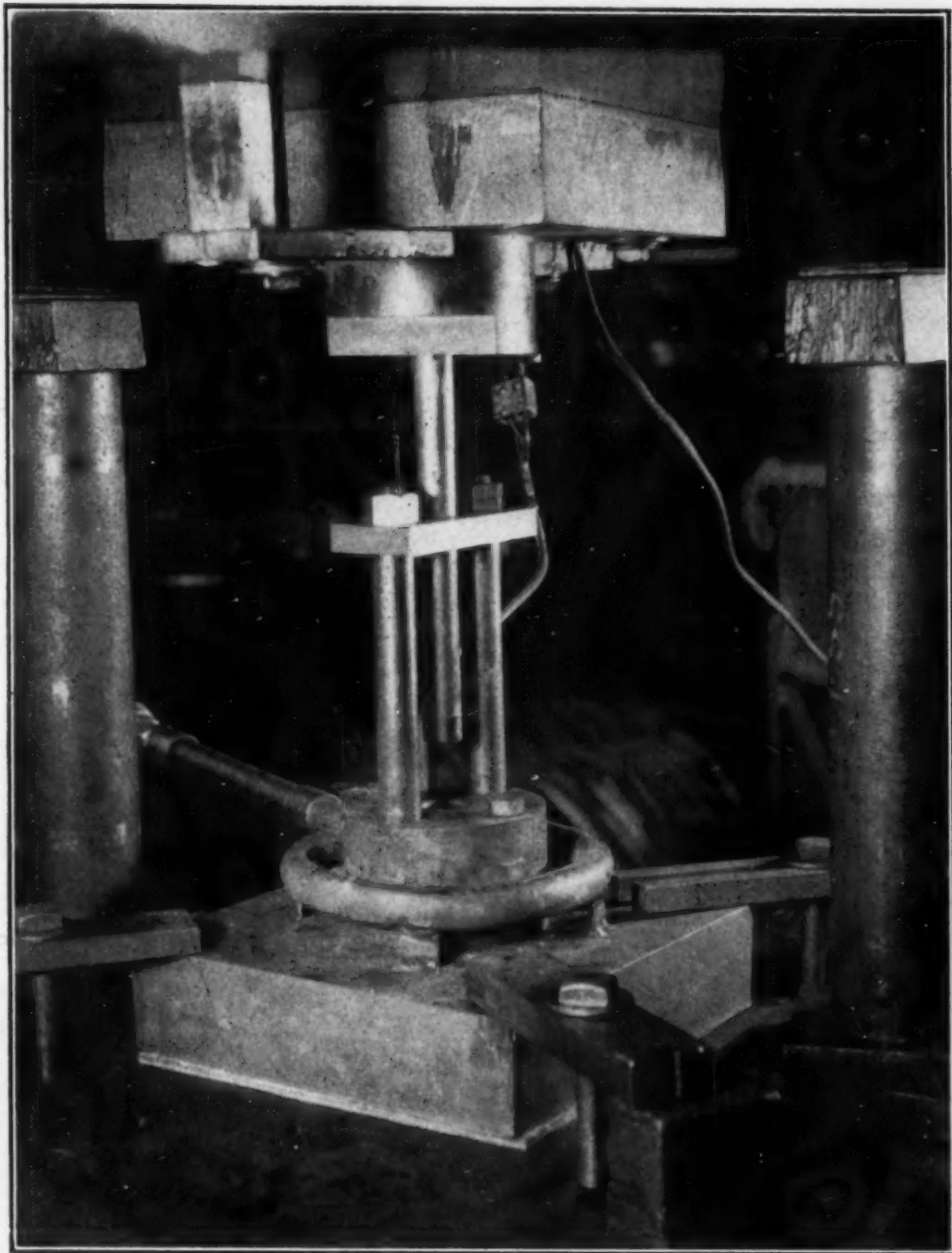


Fig. 19—Back Extrusion Die.

The threaded portion on the base of the cup at the left was formed with a $\frac{1}{4}$ -20 tapped hole in a $\frac{1}{2}$ -inch thick steel plug placed in the bottom of the shaper. The threaded plug was unscrewed from the cup after the extrusion was completed. The other cup was made in the same manner with the exception that a $\frac{1}{2}$ -13 tapped hole was used instead of the smaller one.

PART II

JOINING METHODS

The purpose of the following discussion is to review the various methods used to join magnesium alloy parts and to discuss briefly the processing involved in fabricating structures by these

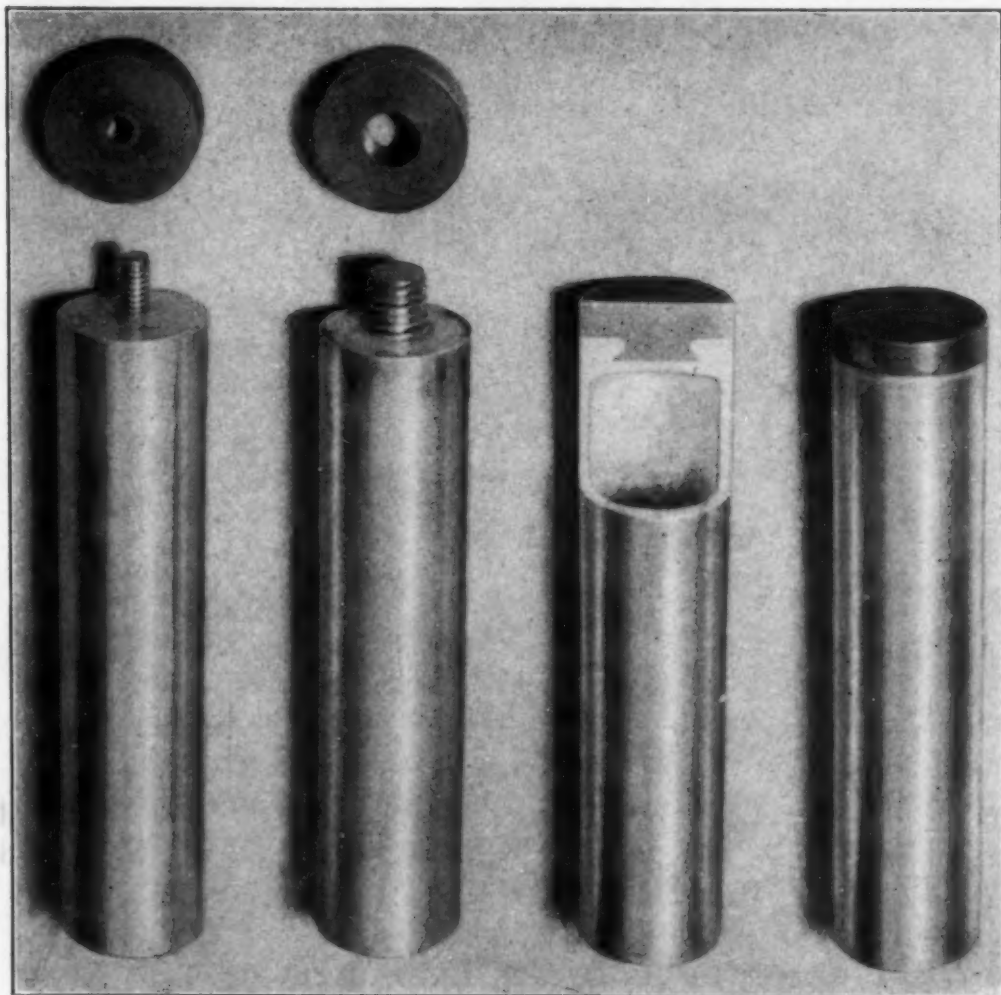


Fig. 20—Back Extruded Cups Combined With Forging Operations.

methods. The equipment and technique necessary to make a given type of joint will not be covered in detail unless the information is new or its inclusion is necessary to preserve the continuity of the material being presented.

SPOT WELDING

Spot welds are used mainly in low stress applications and in high stress applications not subject to excessive vibration. Spot

welds have been used on primary aircraft structures without trouble and their use for this purpose will probably become general after additional service experience is obtained. The success of spot welding as used in secondary aircraft structures is evidenced by the fact that thousands of spot welded parts are giving satisfactory service.

All sheet alloys and extrusion alloys can be spot welded. In welding different alloy sheets or extrusions to each other the ease of welding depends on the alloying constituents in the materials; the more identical the composition of the materials, the easier they are to weld. Materials with different alloying elements, for example J-1 alloy and M alloy, may be welded to each other only with difficulty as very careful control of welding conditions is required.

Both A.C. and D.C. welders of the type used in spot welding aluminum alloys have sufficient capacity and provide adequate control of current and electrode force to make good welds in magnesium alloys. While fairly good welds can be made with welders of the type usually used for ferrous metal welding, these machines normally are not provided with accurate enough current and electrode force controls to spot weld structures subject to rigid specifications such as those enforced in aircraft work.

Areas to be spot welded should be cleaned free of pickle coatings or of oxidized surfaces on unpickled sheet. At present cleaning is usually accomplished by wire brushing both sides of the sheet in the area to be welded with a brush of steel wire 0.003 to 0.006 inch in diameter rotated at a peripheral speed of over 2500 feet per minute. This is followed by rubbing the side of the sheet to be contacted by an electrode with No. 3 steel wool or with No. 160 to No. 240 aluminum oxide cloth. Small areas may be cleaned by hand using only steel wool or abrasive cloth. Stainless steel wool is preferred because of its nonmagnetic properties. Aluminum oxide cloth is used as it results in minimum surface contamination.

Chemical cleaners for spot welding have been tested which show promise of being superior to mechanical cleaning methods, both from the standpoint of cost and thoroughness of cleaning action, but all development work is still in the experimental stage.

The same types of electrodes as those used in welding aluminum alloys are recommended for magnesium alloys. The most satisfactory electrode contour has been found to be a 2 to 8-inch spherical radius. Water-cooled electrodes with an intermittent water supply so that water is flowing only while the welding operation is in prog-

ress are usually employed so temperatures may be held at a constant level. The tip temperature generally used in welding all materials is one which is as low as possible yet does not result in condensation on the electrodes.

In setting up a machine the proper pressure, current, electrode contour, etc., should be selected which will result in a correct diameter spot as determined by strength requirements and which will have required penetration. In making the set-up, care should be

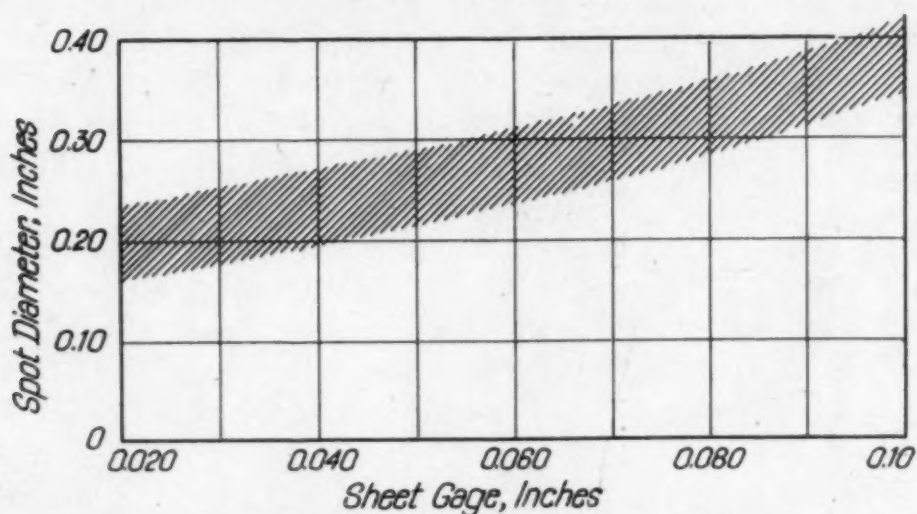


Fig. 21—Recommended Spotweld Diameters M, J and FS Sheet.

taken to make sure the welds are free from spitting, cracking, or porosity. A curve showing the range of spotweld diameters in all alloys recommended for typical structures is given in Fig. 21.

The number of spots which may be made without tip cleaning varies from 10 to 20 spots for M alloy, 30 to 50 spots with FS-1, and from 100 to 150 spots on J-1. While tests have proven that clean spot welds are not selectively attacked in corrosive surroundings, copper pickup will cause corrosion and care must be exercised to avoid pickup. Copper deposit is detected as a black discoloration when the spot welded part is chrome pickled or by placing a drop of 10 per cent acetic acid solution on the spots. Welds showing evidence of copper pickup should be cleaned with steel wool or with an aluminum oxide abrasive cloth.

It is recommended that all spot welded parts be given a chemical treatment incorporating a dichromate boil such as Dow No. 7 treatment for alloy FS-1 and J-1 and Dow No. 10 treatment for M alloy.

As all the semi-liquid materials used to protect faying surfaces in spot welded joints are adversely affected by a dichromate boil they generally are not used on magnesium alloys. If a protector is used a treatment not involving a boil must be used and it has been found the omission of a boil treatment is more deleterious to the corrosion resistivity of a structure than is the omission of the interface protection for practically all service conditions.

RIVETING

Riveting, in magnesium alloys as in aluminum alloys, is still the most widely used joining method for general assembly work. Rivets are used in magnesium alloys in much the same way as in aluminum alloys and only a few minor changes in technique are required.

56S aluminum alloy rivets are most suitable for magnesium alloys. This alloy has a low potential in contact with magnesium alloys and effectively prevents galvanic corrosion troubles. 56S rivets are not heat treatable and should be purchased in the temper required. 56S-1/4h rivets are recommended except where the rivet is to be used in dimpled sheet in which case 56S-O rivets are normally used. No precautions need be taken to insulate this material from magnesium. In cases where assembly conditions are of a nature which would make it difficult to drill the rivet holes and machine countersink the sheet before chemically treating and priming, the rivet is often used in direct contact with the magnesium without trouble. These rivets are always anodized to provide better paint adhesion. The use of rivets other than 56S alloy will be discussed later in this section. Steel or brass rivets, fittings, bolts, nuts, etc., should not be used on magnesium structures unless they are cadmium or zinc plated and insulated with zinc chromate compound or other sealing material.

Drilling and piercing methods are both used to make rivet holes in magnesium alloy sheet. Drilling is preferred for highly stressed parts subject to fatigue as piercing results in a flaky edge, especially in hard-rolled sheet thicker than 0.04 inch. While standard 118-degree point angle drills can be used for drilling sheet satisfactorily, better results can be obtained by using the same drill sharpened to a 60 to 80-degree included angle with the web thinned and the corners rounded. Standard countersink tools used for aluminum alloys are also employed for magnesium alloys.

Standard rivet squeezers and pneumatic hammers used in riveting aluminum are both satisfactory for magnesium alloys. The choice of equipment is dictated by the nature of the structure being riveted as exhaustive test work has shown little difference in the quality of work produced by either method. Care, of course, must be used with a hammer to drive the rivets squarely and not allow the header to dent the sheet.

The rivet length for a given grip in magnesium is usually made slightly less than for aluminum when using flat aircraft type bucktails as a thorough investigation on the effect of bucktail sizes has proven that full strength joints can be developed with bucktails driven to a minimum height of 0.4 times the rivet diameter and a minimum diameter of 1.33 times the rivet diameter. The correct size bucktail is obtained by selecting a rivet length which projects 1 to 1.25 times the rivet diameter before driving.

Investigations on all sheet alloys have shown that an edge distance of 2.5 times the rivet diameter (measured from the center of the hole to the edge of the sheet) is adequate to prevent cracking or bulging in the edge of the sheet when the rivet is driven and gives maximum strength joints. The above was checked using sheared sheet edges which presents the most unfavorable condition from both driving and strength considerations.

Tests have been made to establish the minimum sheet gage in which machine countersunk rivets may be used and the limits arrived at are given in Table VI.

Table VI
Minimum Gage Sheet for Machine Countersunk Rivets

Rivet Diameter	Recommended Minimum	Absolute Minimum
$\frac{1}{8}$	0.040	0.032
$\frac{3}{16}$	0.051	0.040
$\frac{1}{4}$	0.064	0.051
$\frac{5}{16}$	0.081	0.072

Where flush rivets are required for sheet thinner than the recommended minimum listed above it is advisable to use press countersunk joints if high strength is required. The main difference in making dimples in magnesium alloy sheet is that heat is usually used to prevent cracking and to forge the material as required to obtain good dimple fit-up and a flush surface.

At present dimples are formed with dies heated by contact with

die holding blocks provided with electric resistance heaters. Thermocouples or sensitive bulbs for temperature controlling instruments should be mounted in the heated die holders as close as possible to the dies. The sheet to be dimpled is heated by contact with the dies rapidly enough to allow dimpling rates of 15 to 30 per minute in production runs. Dimpling may be accomplished satisfactorily in hydraulic or pneumatic machines which have sufficient throat to handle the die holders and which develop adequate pressures to form the dimple properly. A machine with a capacity of 5000 pounds will handle all size dimples up to $\frac{3}{16}$ inch in diameter in 0.072 sheet. Fig. 22 shows a pneumatic arbor press set up with a bottom die holder suitable for dimpling parts such as rib flanges which require clearance directly under the point of dimpling. The lower holder is heated by means of three 300-watt strip heaters clamped to the lower side of the plate. The upper die holder is heated by means of a hollow cartridge heater in this case. Higher heats can be maintained more satisfactorily, however, by clamping four 300-watt strip heaters to a square piece of steel machined to fit the arbor at one end and to hold the die on the other end. In operation the sheet is located on a pin in the upper die and the area of sheet to be dimpled allowed to preheat. In starting a new row of dimples a few seconds' pause is required before the press stroke is started but subsequent dimples may be made almost as fast as the sheet can be located on the pin as a certain amount of preheating is effected by forming the first dimples.

Die temperatures between 450 and 550 degrees Fahr. (230 and 315 degrees Cent.) have been found satisfactory for all sheet. Checks have shown that the sheet being dimpled will reach a temperature approximately 50 degrees Fahr. below the die temperature in the few seconds required for making a dimple. Temperatures above the annealing point may be used on hard-rolled sheet as the total time the sheet is above critical temperature is only 4 or 5 seconds. Dimples can be formed with the dies held lower than 450 degrees Fahr. (230 degrees Cent.) but the rate of dimpling is slower due to the lack of sufficient thermal head to reach the required temperature rapidly.

Highest strength dimples in magnesium alloys are obtained by forming the dimples with a pilot hole approximately 15 per cent smaller than the rivet and redrilling to the required size after the dimple is formed. A sharp edge should not be left on the bottom of

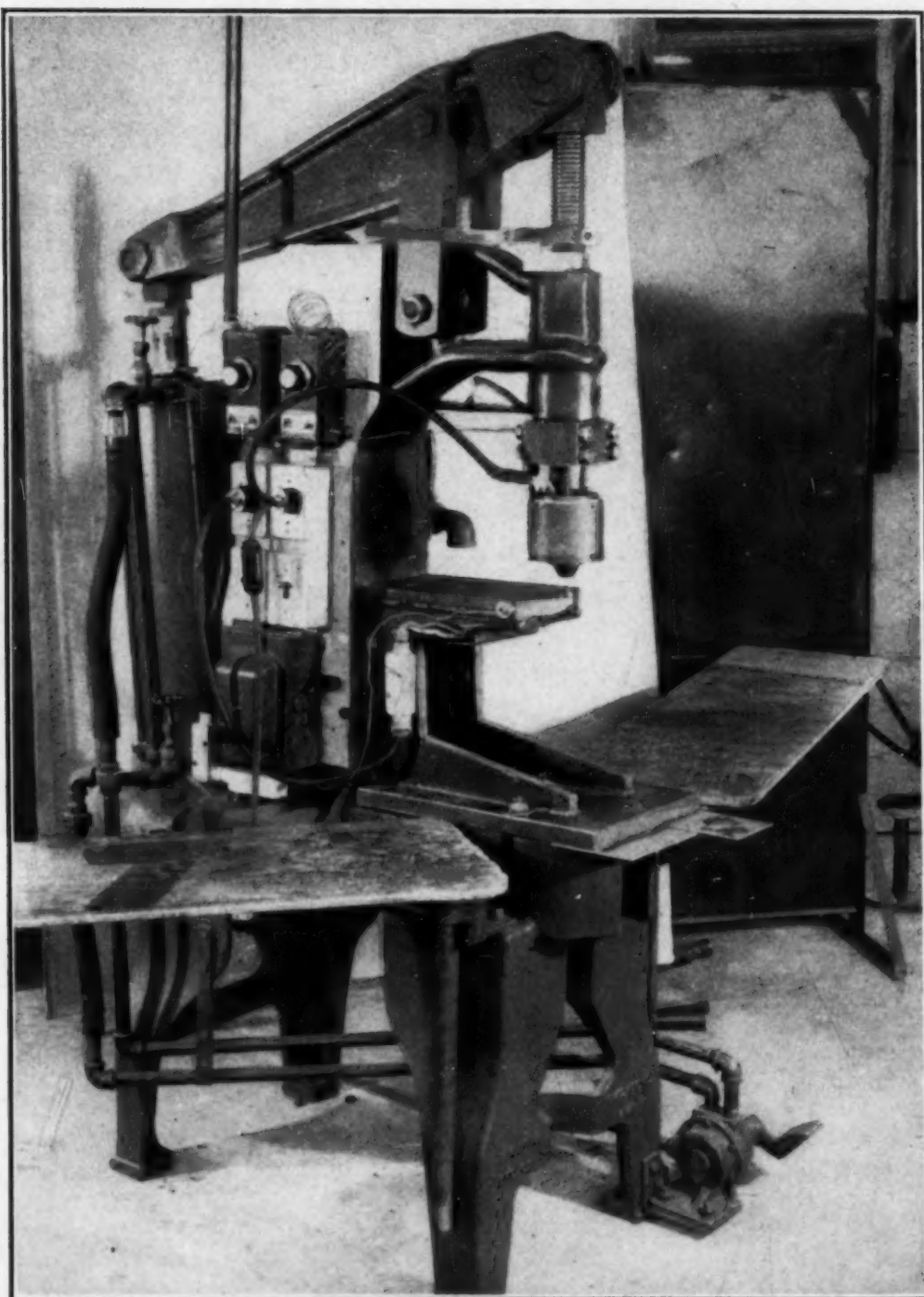


Fig. 22—Press Fitted for Hot Dimpling Magnesium Alloy Sheet.

the dimple after the redrill operation as it may cause cracks when the rivet head is formed. Flats are provided on the dimple against which the rivet bucktail is formed by forging a flat at the time the dimple is formed or by removing the edge with a file or other cut-

ting tool after the redrill operation. The diameter of the flat provided should be from 1.33 to 1.5 times the rivet diameter. This flat should be placed only on the sheet against which the bucktail is formed. Placing the flat on the dimple which nests in the bottom dimple leaves a gap in the material which lowers the strength of the joint and which may cause the bottom dimple to break when the rivet is headed.

Up until fairly recently it was necessary to use 120-degree included angle rivets in J-1h sheet when press countersinking was required to eliminate cracking experienced when 100-degree rivets were used. Dimpling technique, however, has recently been refined and present indications are that standard 100-degree included angle flush rivets may be used in all magnesium alloy sheet without trouble.

Fig. 23 shows a wing constructed from J-1h alloy sheet and O-1HTA extrusions. One-eighth-inch flush rivets are used in the skin forward of the front spar. Machine countersinking is used in the 0.051 skin on about the inner third of the panel and press countersinking is used on the outer $\frac{2}{3}$ of the panel where thinner skin than 0.051 is employed.

The following procedure is suggested as a method for processing typical riveted assemblies where 56S alloy rivets are used.

1. Cut blank, jig drill pilot holes, and form block locating pin holes where required. Form and if necessary trim parts.
2. Apply chemical treatment called for on finished assembly.
3. Parts inspection.
4. Paint parts with zinc chromate primer. Note—Omit this step if parts are to be dimpled.
5. Store parts.
6. Drill, countersink, and dimple as required. Note—Dimpled parts are painted with zinc chromate primer after dimpling.
7. Rivet.
8. Rivet inspection.
9. Bucktails primed with zinc chromate either locally or by priming entire bucktails side of structure.
10. Finish painting.
11. Final inspection.

When 2S, 3S, or A17ST alloy rivets are used the above pro-

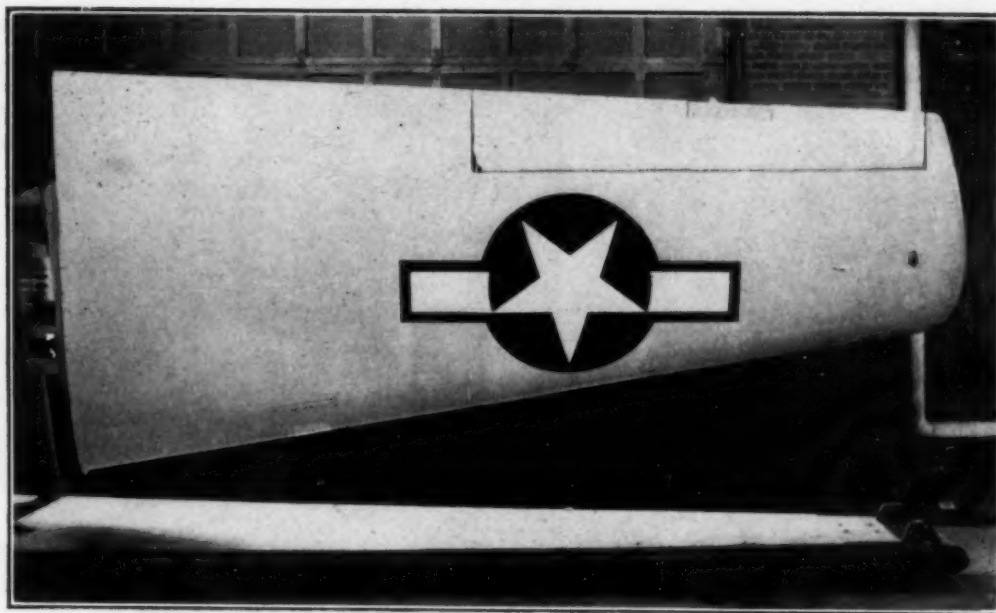


Fig. 23—Wing Made from J-1h Alloy Sheet and O-1HTA Extrusions.

cedure may be followed but the rivets must be set in wet zinc chromate primer.

If 17S or 24S alloy rivets are used the procedure must be changed so all drilling, machine countersinking, and dimpling operations are performed before the parts are given a finish chemical treatment. These rivets must be set in wet primer in addition to having all surfaces in contact with the rivet pickled and primed.

GAS WELDING

The advisability of fabricating a structure by means of fusion welding in preference to riveting, spot welding, etc., is usually dictated by the shape of the structure and the purpose for which it is intended. The decision on whether to use gas welding or arc welding is more difficult to make although in some cases either method is equally satisfactory. General rules limiting the use of either method will be given in this section and that following on arc welding; the advantage of each over the other is listed in the respective sections.

Gas welding is usually preferred to arc welding for light gage sheet parts (less than 0.1 inch thick) which are not highly stressed. Fit-up is not critical for gas welding; for example, it is possible to weld parts together where gaps as large as six times the material thickness (up to a maximum of $\frac{3}{8}$ inch) occur. This is a definite

advantage in fitting up complicated formed parts where the accurate fit-up required for arc welding is difficult to obtain. The closer fit-up for arc welding requires the use of fairly elaborate jiggling whereas gas welding is accomplished with the minimum jiggling required to hold the parts in proper alignment. The consensus of opinion is that gas welding requires less technique than arc welding on the part of the operator and is therefore somewhat easier to use. The cost of arc welding is slightly higher than gas welding due to the present price level on inert gases. This, however, is offset to some extent by the flux removal operations necessary for gas welding. It is not necessary to stress relieve gas-welded M alloy assemblies. This is required in arc-welded assemblies to eliminate the danger of locked-up stresses. Gas-welded alloy J-1 and FS-1 assemblies should be stress relieved the same as arc welds. Stress relief methods will be discussed in the following section on arc welding.

Alloy M is readily gas weldable. Gas welds in FS-1 and J-1 must be limited to free welds, otherwise cracking is liable to occur. Gas welding is preferably limited to joining like alloys although successful welding may be done if the materials have the same alloying constituents and their melting points are nearly equal. When cast fittings are welded to wrought structures the casting should preferably be made from the same alloy as the wrought material.

Gas welds are limited to butt type joints or corner joints which do not result in flux entrapment such as occurs in lap joints, edge joints or plug type welds. This requirement must be strictly adhered to as the flux used in welding is an active corroding agent in the presence of moisture and when left in contact with magnesium will invariably result in trouble. If Tee welds are made by gas welding, the weld should be made from one side and penetration should be sufficient to push the flux from between the abutting surfaces. All joints should be limited to gages which can be welded in a single pass as flux entrapment is apt to occur when a weld is reflowed for multiple pass welds. The maximum sheet thickness which can be butt welded in a single pass and which therefore limits the maximum gage sheet which should be gas welded is 0.25 inch.

Standard lightweight aircraft type welding torches are used for welding magnesium alloys. Tip sizes with orifices ranging from 0.035 to 0.081 inch are used depending on the gage sheet being welded.

Various gases and mixtures of gases are used with oxygen for

welding magnesium alloys. Acetylene gas is satisfactory for welding gages heavier than 0.064 but normally is not used on thinner sheet as the flame is too hot and therefore difficult to control. Acetylene also causes pitting on the weld bead surface which is probably caused by the effect of its high temperature on the flux. The pitting is not particularly harmful but it does give an inferior appearance. Carbohydrogen gas (a mixture of hydrogen and methane) is ideally suited for welding magnesium alloys, particularly in the thinner gages, but is difficult to obtain in certain parts of the country. It has a lower flame temperature than acetylene and is very easily regulated. If this gas is used in welding heavy sheet small amounts of acetylene are sometimes mixed with it to increase the flame temperature. Mixtures of hydrogen and acetylene are also used satisfactorily and offer a means of welding light sheet when carbohydrogen is not available. Unmixed hydrogen is not usually used because of the difficulty of adjusting the flame. In using all of the above gases the torch should be adjusted so the flame is neutral or slightly reducing.

The flux used to prevent oxidation while welding may be obtained from manufacturers of magnesium products. Dow No. 460 flux is recommended for welding with all types of gases. A second flux, Dow No. 450, is also available but it is suitable only for oxyacetylene. Even though No. 450 flux results in more surface pitting than No. 460 flux, some users prefer it because it has a higher fluidity than No. 460 flux, it being similar to aluminum welding fluxes in this respect. These fluxes are in powder form when purchased and are mixed in the proportion of one part water to two parts powder by volume to obtain a paste suitable for brushing on the weld area. As the fluxes are hygroscopic they should be stored in closed glass bottles until ready for use.

Various diameter extruded welding rod is available in all alloys. The rod should be of the same alloy as the material being welded but in case it is not available an alloy with slightly lower melting point may be used. If it is necessary to weld different alloy materials together, rod having the composition of the lower melting point material should be used. The recommended range of sheet gages on which a given size rod may be used is listed in Table VII.

All grease, oil, dirt, and other foreign matter should be cleaned from parts to be welded by means of solvents or alkaline cleaners. Unpickled sheet may be welded without further cleaning but sheet

Table VII
Rod Size Versus Sheet Thickness

Rod Diameter Inches	Sheet Gage Inches
$\frac{3}{16}$	up to 0.064
$\frac{1}{8}$	0.051 to 0.128
$\frac{3}{32}$	0.091 to 0.25

which has been chrome pickled should be cleaned free of the coating by means of steel wool or a wire brush. Pickled sheet is sometimes welded without cleaning but the chromates cause porosity in the weld metal and impair the free flowing characteristics obtained with bare surfaces. Sheet protected with a special "in process" pickle applied by a 1-minute dip in a bath composed of 15 to 20 per cent chromic acid and 3 per cent of calcium, sodium, potassium, or magnesium nitrate (this is the same bath used to remove graphite from formed parts) can be welded as well as bare material. This treatment may be used to protect parts in storage and serves as well as a chrome pickle as a protection from corrosion. (Note: This pickle is not used as a finish treatment due to the poor paint adhesion it affords).

Sheet up to $\frac{1}{8}$ inch in thickness can be welded with square edges. Sheet over $\frac{1}{8}$ inch should be beveled on one side to form an included angle of approximately 90 degrees. The vee should not extend through the sheet but should have about a $\frac{1}{16}$ -inch flat or land remaining to provide better fit-up conditions and to eliminate the sharp edge from melting away as soon as heat is applied.

Jigs should be provided to hold the sheet in good alignment for tack welding. The usual practice is to remove an assembly from a jig after tack welding and finish welding without a jig. Sheets of the same gage up to a thickness of 0.064 inch are normally butted flush for welding. Sheets from 0.064 to $\frac{1}{8}$ inch thick are set up with a gap approximately equal to the metal thickness being welded. Beveled sheet from $\frac{1}{8}$ to $\frac{1}{4}$ inch thick should be set up with a gap of $\frac{1}{16}$ inch. When sheet thinner than 0.064 is butted to sheet thicker than 0.064 no gap is provided.

Cast fittings or fittings machined from extruded stock are preferably provided with a flange about the same thickness as the sheet into which they are to be welded to eliminate trouble with the sheet melting away before a heavy fitting section could be heated to the temperature required for fusion. Parts of a weld jig for lo-

cating fittings should be made adjustable in a direction perpendicular to the surface of the sheet in which a fitting is to be welded. This is necessary because the sheet edge may move in or out when it is heated, depending on the shape of the part, and the fitting must be so held that while the welding is being accomplished good line-up of the fitting flange and sheet may be obtained. For example in welding a fitting in the end of a 14-inch hemispherical tank head it is necessary to set the casting about $\frac{3}{32}$ inch above the sheet surface when the head is cold to allow for expansion during welding. After the proper location to obtain alignment is determined experimentally the casting locator is locked in place.

Parts to be welded are fluxed on both sides of the sheet at the point tack welds are to be placed, the distance between tacks being from 1.5 to 6 inches apart, depending on the shape of the parts being welded and the gage sheet being used. If the welding rod surface is dirty or badly oxidized it should be cleaned with steel wool. The rod is fluxed and the tacks are made after which the weld area on both sides is fluxed and the joint completed. In starting the weld the torch flame is moved over the point at which the bead is to be started until the flux has been thoroughly dried. The actual manipulation of rod and torch is very similar to that employed in welding aluminum, in fact welders familiar with aluminum have made good welds in magnesium alloys the first time they tried to weld it. If an incompletely fluxed area starts to burn while the bead is being run the torch should be removed and the burning will stop immediately. The burned area should be scraped clean and refluxed before the weld is continued.

On completion of a bead the torch should be played on the work for a few seconds to allow the stopping point to cool slowly. If this is not done the rapidly cooling weld metal may contract locally and result in a small pit or crater.

Fig. 24 shows a cast fitting being tack welded in assembling an M alloy tank. The jig being used locates all fittings accurately with respect to each other. After all fittings are tacked in, the assembly is removed from the jig and the castings are welded in place. After the part is welded all traces of flux should be removed from accessible areas by scrubbing the weld area with hot water and a steel wire brush. Normally this operation may be postponed up to an hour after welding when No. 460 flux is used. As this flux is hygroscopic and corrosion will appear rapidly when it absorbs moisture,

however, on days when the humidity is high the cleaning operation should be accomplished as soon as possible after the weld is made. No. 450 flux, as well as other fluxes other than No. 460 flux which have been used, are so hygroscopic that immediate washing is necessary regardless of humidity conditions. Following the scrubbing the parts are given a No. 1 treatment or an "in process" pickle depending on the alloy and final treatment the part is to have. If a part is to

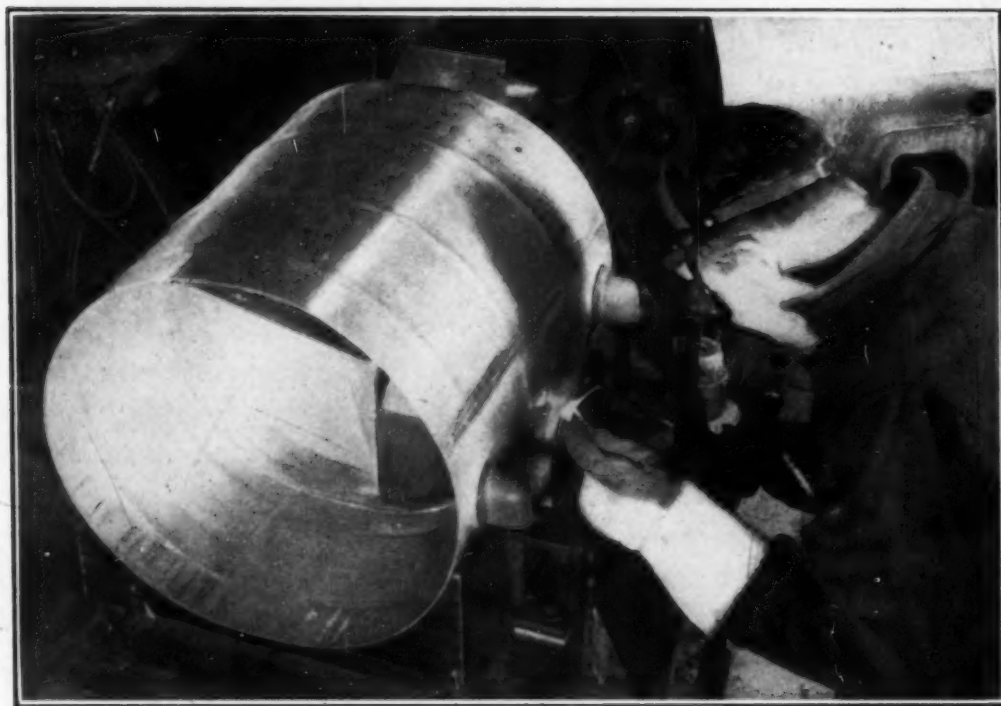


Fig. 24—Gas Welding Operation on a Ma Alloy Oil Tank.

be finished with a No. 1 or a No. 10 treatment the No. 1 treatment is used providing further welding is not required. If the part is a sub-assembly and further welding is required or if it is to receive a No. 7 treatment the "in process" pickle is used. As mentioned before the "in process" pickle need not be removed for welding. The No. 1 treatment does not detract from the protection afforded by a No. 7 treatment but it does change its appearance. The "in process" pickle will not affect the No. 7 treatment in any way.

After the assembly has been completely welded and pickled it should be given a 1 to 2-hour boil in a 5 per cent solution of sodium dichromate to effect complete removal of any surface flux and to provide additional surface protection. This boil serves to remove flux which cannot be removed in the scrubbing operation as, for

example, when a head is attached in finishing a tank assembly. It is used only after an assembly has been completed and is not necessary on sub-assemblies to be stored except for removal of flux from inside seams which cannot be scrubbed.

ARC WELDING

The use of arc welding in preference to gas welding is often dictated by the type of joint required. While gas welding must be limited to butt type joints to insure removal of the corrosive flux used, arc welding, which utilizes an inert gas shield to prevent oxidation of the material instead of a flux, can be used on lap joints, fillet joints, and all other joints such as those commonly used in steel arc welding. As multipass welds are permissible there is no top limit on the material thickness which may be welded as there is in gas welding. Generally arc welding is preferred to gas welding in material over 0.1 inch thick due to the faster welding rate obtainable.

Arc welding is preferred for parts where warpage must be held to a minimum. The reduced warpage is effected by the higher heat available with the arc which enables fusion temperatures to be reached with less diffusion of heat to adjacent metal.

Arc welding is employed where maximum strength joints are required in alloy M and J-1 structures. The strength of gas-welded FS-1 sheet is approximately the same as when arc welding is used. A comparison of the efficiencies which may be obtained with butt welds made by both methods is given in Table VIII.

Table VIII
Efficiency of Gas and Arc Welds

Alloy	Gas Weld*	Per Cent	Arc Weld*
	Efficiency		Efficiency
J-1	90		95
M	65		70
FS-1	90		90

*Efficiency with bead as welded. If bead ground flush the efficiencies will be 5 to 10 per cent lower.

All wrought materials except FS-1 sheet have good arc weldability. FS-1 sheet has a limited application for arc-welded structures, but as in gas welding, restrained welds must be avoided or cracking may result.

In arc welding magnesium alloys the arc is struck between a tungsten electrode and the work much in the manner used in the

carbon arc welding of steel. While welding is in progress the arc is enveloped in an inert gas shield which excludes oxygen from the weld area and therefore prevents oxidation of the heated metal. This inert gas is applied by flowing it into the weld area from a cup surrounding the electrode. The function of a torch for arc welding magnesium alloys is to hold the electrode and cup and to carry the welding current and shielding gas. A valve is provided in the torch handle to control the flow of inert gas to the work.

Both helium and argon have been found satisfactory for shrouding the arc, the choice of gas depending largely on the price of each and the type of welding for which it is to be used. It has been determined that about three times more helium by volume than argon is required for a given amount of welding; therefore, once the cost per cubic foot of each is determined the price advantage can be readily computed. Helium does not require as close an arc length as argon, therefore it is somewhat easier for a welder to use. The automatic voltage regulator on an automatic welder does not function when argon is used. It must be disconnected and the weld made with a set arc length. When helium is used the regulation is satisfactory and fully automatic welding may be accomplished.

D.C. and rectified A.C. arc welding machines of 100 to 200-ampere capacity are normally used for inert gas arc welding of magnesium alloys. The machine should be of the stable arc type and should be equipped with a continuous ampere regulator to provide adequate current control. Reversed polarity (electrode positive and work negative) is required as straight polarity results in a wild arc unsuitable for welding. Use of a straight A.C. machine also results in a wild arc.

Various materials have been tried for electrodes but tungsten is the only one which has proven entirely satisfactory. Electrodes ranging in size from $\frac{3}{32}$ to $\frac{1}{2}$ inch in diameter are used. The electrode should extend from $\frac{1}{4}$ to $\frac{3}{8}$ inch beyond the end of the gas cup for normal welding. It may be extended beyond $\frac{3}{8}$ inch to reach tight corners. The length should not exceed $\frac{3}{8}$ inch except for special cases, however, or excessive gas will be required to maintain a proper shield.

The filler rod should preferably be of the same alloy as the material being welded. Sizes of filler rod ranging from $\frac{3}{32}$ to $\frac{1}{4}$ inch in diameter are adequate for making welds in any size material.

Accurate control of inert gas flow is obtained by means of an

oxygen therapy regulator or a flowmeter used in conjunction with a standard oxygen or hydrogen regulator. The reading obtained with a flowmeter designed for giving oxygen flow in liters per minute can be converted to helium flow in cubic feet per minute by dividing the reading by 10.

Table IX gives data required for arc welding various gages of magnesium alloy sheet.

In preparing sheet for welding it is essential that all oil, grease, chemically treated surfaces, oxide films, and other impurities be removed from the weld area. This may be accomplished by use of solvents and steel wool or by scraping. Where a weld is made from one side of a sheet the oxide coating on plain sheet or the pickled surface on a chemically treated sheet should not be removed from the bottom side of the sheet as these surfaces are beneficial in controlling the dropdown of the weld.

Sheet up to 0.1 inch thick is welded with a square butt joint. Material from 0.1 to $\frac{3}{16}$ inch thick is beveled from the welding side to about a 90-degree vee which extends sufficiently far into the sheet so a $\frac{1}{16}$ -inch land or flat remains. Sheet over $\frac{3}{16}$ inch thick should be welded from both sides wherever the nature of the structure permits as sounder welds may be obtained and less warpage results. For a double vee joint the 90-degree included angle should extend from both sides so as to leave a $\frac{1}{16}$ -inch land in the center of the sheet. When welding a double vee joint the back of the first bead should be chipped out using a chipping hammer fitted with a cape chisel (a rotary file may be used but the chipping hammer is much faster) to remove oxide film, dirt, and incompletely fused areas before the second bead is added, if maximum soundness is to be obtained in the weld. Chipping of the back side of a weld is also used in some cases where welds are made from one side in heavy material for the same reasons as those listed for a double vee weld.

In making Tee welds the leg of the Tee is not veed if it is under 0.080 thick. Thicker material should be single or double beveled to an open angle of 60 degrees, leaving about a $\frac{1}{32}$ -inch land at the base. Single bevels are used on material up to $\frac{3}{16}$ inch thick to be continuously welded and double bevels used where material over $\frac{3}{16}$ inch thick is to be continuously welded. Where stagger tack weld Tee joints are used and the leg of the Tee is over 0.08 it is beveled to a 60-degree angle halfway through the sheet at the points the tacks are to be placed.

Table IX
Arc Weld Data

Thick- ness Inches	Current Amps. ¹		Filler Rod Diameter Inches	Helium Flow Cu. Ft./Min.	Tungsten Electrode ² (Diameter in Inches)	
	Alloys J-1, FS-1 and O-1	Alloy M			J-1, FS-1 and O-1	M
0.030	15- 25	20- 30	$\frac{3}{32}$	0.3 to 0.6	$\frac{3}{32}$	$\frac{3}{32}$
0.040	20- 30	25- 35	$\frac{3}{32}$	0.3 to 0.6	$\frac{3}{32}$	$\frac{3}{32}$
0.050	20- 35	30- 45	$\frac{3}{32}$	0.3 to 0.6	$\frac{3}{32}$	$\frac{3}{32}$ or $\frac{1}{8}$
0.060	30- 45	40- 55	$\frac{3}{32}$ or $\frac{1}{8}$	0.3 to 0.6	$\frac{3}{32}$ or $\frac{1}{8}$	$\frac{1}{8}$
0.070	45- 60	50- 65	$\frac{1}{8}$	0.3 to 0.7	$\frac{1}{8}$	$\frac{1}{8}$
0.080	55- 70	65- 80	$\frac{1}{8}$	0.3 to 0.7	$\frac{1}{8}$ or $\frac{7}{32}$	$\frac{1}{8}$
0.090	60- 75	70- 85	$\frac{1}{8}$	0.3 to 0.7	$\frac{1}{8}$ or $\frac{7}{32}$	$\frac{1}{8}$
0.100	70- 90	80-100	$\frac{1}{8}$	0.4 to 0.8	$\frac{7}{32}$	$\frac{1}{8}$
0.125 ³	75-100	85-110	$\frac{1}{8}$ or $\frac{3}{16}$	0.4 to 0.8	$\frac{7}{32}$	$\frac{1}{8}$
0.150	80-110	90-120	$\frac{3}{16}$	0.4 to 0.8	$\frac{7}{32}$	$\frac{7}{32}$ or $\frac{1}{4}$
0.200 ⁴	70- 90	80-100	$\frac{3}{16}$	0.4 to 0.8	$\frac{7}{32}$	$\frac{7}{32}$
0.250	80-100	90-110	$\frac{3}{16}$ or $\frac{7}{32}$	0.4 to 0.8	$\frac{7}{32}$	$\frac{7}{32}$
0.500 ⁵	90-120	100-130	$\frac{3}{16}$ or $\frac{7}{32}$	0.5 to 1.0	$\frac{7}{32}$ or $\frac{1}{4}$	$\frac{7}{32}$ or $\frac{1}{4}$
0.750 ⁶	110-140	120-150	$\frac{7}{32}$ or $\frac{1}{4}$	0.5 to 1.0	$\frac{1}{4}$	$\frac{1}{4}$
1.000	120-150	130-160	$\frac{7}{32}$ or $\frac{1}{4}$	0.5 to 1.0	$\frac{1}{4}$ or $\frac{3}{8}$	$\frac{1}{4}$ or $\frac{3}{8}$

¹Currents given are for welding speeds of approximately 12 inches per minute in single pass welds. In multiple pass welds the welding speed per pass is higher.

²The inside diameter of the inert gas cup should correspond to the tungsten diameter as follows: $\frac{1}{8}$ - and $\frac{7}{32}$ -inch electrode— $\frac{1}{8}$ -inch cup. $\frac{3}{16}$ -inch electrode— $\frac{1}{4}$ -inch cup. $\frac{1}{4}$ -inch electrode— $\frac{3}{8}$ -inch cup. $\frac{3}{8}$ -inch electrode— $\frac{1}{2}$ -inch cup.

³Sheet 0.1 to $\frac{1}{8}$ inch thick beveled from one side of sheet.

⁴Sheet over $\frac{1}{8}$ inch thick beveled from both sides of sheet.

⁵Multiple pass welds in sheet over $\frac{1}{4}$ inch thick.

⁶In welding material $\frac{1}{4}$ inch thick or heavier it is desirable to preheat the material to 500 to 600 degrees Fahr. (260 to 316 degrees Cent.) to increase the welding rate.

Jigs for arc welding are usually more complete than those used in gas welding. The entire welding operation is normally completed in the weld jig instead of being removed from the jig after tack welding as is the case in gas welding. Fit-up for welding must be quite accurate as gaps in the sheet make it difficult to shield the weld properly. The function of an arc welding jig is to hold the parts being welded in proper alignment, to keep the weld gap as small as possible, to hold the parts in the proper relative location to each other, and to keep warpage to a minimum.

When parts are properly jigged the bead is run directly without tacking the sheet prior to the operation. Tacks, of course, must be used when jigs are not furnished to hold the sheet in the correct position for welding.

In starting a weld the correct machine settings, inert gas flow, etc., are set as shown in Table IX. The inert gas valve is opened before the arc is struck. After the metal under the arc becomes molten the filler rod is fed to the arc, the actual manipulation of the torch and rod being quite similar to that used in gas welding. The torch should be held as nearly perpendicular to the sheet as possible in order to obtain the maximum shielding effect with a minimum

amount of gas. The filler rod should be held at an angle of at least 60 degrees from the torch to prevent the reflected heat from the electrode causing the rod to melt prematurely and cause an uneven bead and poor penetration.

After parts are welded residual stresses as high as 15,000 pounds per square inch may exist in areas near the weld beads. It is necessary that these stresses be relieved after welding and this may be accomplished readily by heating the structure in accordance with the temperature and time listed in Table X.

Table X
Stress Relief Data for Arc-Welded Magnesium Alloys

Alloy	Temperature Degrees Fahr.	Time at Temperature
FS-1a	500	15 minutes
FS-1h	265	1 hour
Ma	500	15 minutes
Mh	400	1 hour
J-1a	500	15 minutes
J-1h	400	1 hour

Stress relief in the case of FS-1 alloy and even more so in the case of J-1 alloy is essential so that locked-up stresses will not cause stress corrosion cracking. While M alloys are only slightly susceptible to stress corrosion it is believed good practice to relieve the residual stresses to prevent distortion in service and to eliminate any possibility of stress corrosion.

Stress relieving operations are usually accomplished by placing the structure in a jig to prevent distortion during the operation before placing the assembly in an oven. In starting a new job it is advisable to attach thermocouples to the part so the time required to reach stress relieving temperatures can be accurately determined. If steel is used in a jig for stress relieving it must be designed to allow for the greater expansion of the magnesium alloy or the size of the part may be affected. In the case of large structures or where parts of the structure cannot be heated to the required temperature because of other materials in the structure, stress relieving may be accomplished by use of electrically heated metal forms clamped to the weld area.

After stress relieving, the weld area is wire brushed to remove the oxide film left on the bead and the part is ready for chemical treatment. As no flux is used the scrubbing operation and sodium dichromate boil required for gas welding is not necessary.

BONDING CEMENTS

While no actual production of magnesium alloy structures incorporating high strength bonding cements have been made, an extensive program on this material has been run and it is expected it will be placed in use in the near future. Strengths which compare very favorably with those obtained in aluminum alloy joints are reached in magnesium alloy joints.

Virtually the same technique used in applying the cement to aluminum alloys is used for magnesium alloys. The best method of surface preparation prior to coating the surfaces to be bonded is still being studied. Slightly stronger joints can be obtained by lightly wire brushing the surfaces to be joined, followed by an alkaline clean in a boiling 6-ounce-per-gallon solution of sodium metasilicate or other suitable alkaline cleaner, than by applying the cement over chemically treated surfaces. The latter method, however, would be more advantageous from an economic and corrosion protection standpoint. As parts cemented with clean surfaces can be pickled satisfactorily after the joint is made, however, the amount of additional protection afforded by having a treated surface under the bond is questionable.

RESISTANCE FLASH WELDING

This method of joining the ends of bars, tubing, etc., has not been used extensively in joining magnesium alloys although adequate experience has been obtained to prove the process is feasible. Joint efficiencies ranging from 85 to 95 per cent have been obtained in alloys FS-1, J-1, and O-1. It is difficult to obtain good welds in alloy M (probably because of its very narrow freezing range). More extensive work with this material may result in a successful method but experience has indicated it should not be flash welded.

Butt welding (current applied after the pieces to be joined are butted together) is not as successful as flash welding. Higher current is required, it is slower, and the parts to be welded must be more carefully fitted. It also results in hot shortness and the formation of an oxide film between the parts.

Because of the high heat conductivity of magnesium alloys a very rapid "pushup" is required. Best results are obtained with automatic motor operated machines which permit rapid pushup and

give a constant acceleration for maintaining the arc during the flashing period. As magnesium alloys have relatively low electrical resistance high current is required, for example in welding 0.5-inch round bars, from 13,500 to 17,000 amperes are needed.

It is not necessary to clean oxide from the surfaces to be welded or from the surfaces in contact with the clamping die. Care, however, should be taken to remove dirt which might cause burning on a surface held in a clamping die.

CONCLUSION

It is hoped the foregoing discussion will be of assistance to those who have not been intimately associated with the working of magnesium alloys in obtaining a better understanding of the methods used in forming and joining magnesium alloy parts and structures.

Constant development work is being conducted to simplify heating problems encountered in forming operations, to more clearly define the limitations of various metal working methods, to improve joining practice, and in general to establish the technique required to take full advantage of the high strength-weight ratio of magnesium alloys.

DISCUSSION

LESLIE W. BALL:¹ The manufacture of structures from wrought magnesium sheet by means of heliarc welding can be greatly assisted by X-ray studies of sample structures. Our Laboratory's experience has shown that radiography of magnesium welds reveals a great deal of information about the quality and structure of the welds, even more so than in the case of steel welds.

In several cases we have tried X-ray micrographs as a method of examining the structure of welded magnesium products. In addition to the normal information about oxide, etc., this method reveals the distribution of the manganese constituent and thereby provides a method of measuring the extent of some modifications due to heating.

JOHN A. MARDICK:² I would like to ask the author two questions. These are: (1) Which magnesium alloy is best suited to perform the following fabrication operations: forming, drawing, and spinning? (2) Which magnesium alloy is easiest to weld by the heliarc or gas welding method?

Author's Reply

Replying to Mr. Ball, we have had no experience with X-ray micrograph examination of welds in magnesium alloys but we have had considerable ex-

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²Research associate, Revere Copper and Brass Incorporated, Baltimore, Md.

perience with industrial radiography and have found it to be useful in inspection of arc welds, gas welds, and spot welds. We have not observed manganese segregation by means of radiography except in the case of spot welds where the manganese appears as a light ring around the weld.

In answer to Mr. Mardick's first question, Dowmetal Ma and Dowmetal FS-1a are the best magnesium alloys from the standpoint of forming, drawing, and spinning. As mentioned in the text of the paper under a discussion on the various alloys Ma is slightly superior to FS-1a for drawing operations while FS-1a has somewhat better bending qualities.

In reply to Mr. Mardick's second question, both Dowmetal M and Dowmetal J-1 sheet and extrusion may be welded by the helium arc welding method very satisfactorily. There have been some opinions expressed that one alloy welds better than the other but in all cases it was found that the person making the statement was used to working with one of the alloys and not the other and developed preference because of familiarity. FS-1 extrusion may also be arc welded satisfactorily but FS-1 sheet must be restricted to relatively simple welds. Dowmetal M is the best alloy for gas welding.

A NEW METALLOGRAPHIC ETCHANT FOR ALUMINUM BRONZE AND COPPER-BERYLLIUM ALLOYS

By W. C. COONS AND D. J. BLICKWEDE

Abstract

Various recommended etchants were used on aluminum bronze alloys, none of which gave completely satisfactory results. In the course of these trials, a 1 per cent aqueous chromic acid solution used electrolytically was found to give the desired etched surface on aluminum bronzes in the composition range of 10 to 13 per cent aluminum and in all states of heat treatment. No acicular beta or beta prime constituents were encountered in the aluminum bronzes investigated, but it is believed that the proposed etchant would etch such microstructures satisfactorily.

Trials were also made using this etchant on copper-beryllium alloys and excellent results were obtained particularly in that the precipitation hardening stages were clearly delineated in the photomicrographs.

INTRODUCTION

ALUMINUM bronzes are copper-base alloys which usually contain 5 to 14 per cent aluminum and up to 5 per cent iron, manganese, silicon, or nickel. These alloys have found wide use in industry because of their high strength and hardness in the cast state, capacity for hardening and strengthening by heat treatment, exceptionally good corrosion resistance, good retention of hardness, scale resistance at elevated temperatures, good wearing qualities when used with steel, and lower density than other copper alloys. However, to obtain properties of these alloys which meet the specifications of the designing engineer, close control over the chemical composition and the heat treatment is an absolute necessity. The equilibrium diagram for aluminum bronze alloys in the aforementioned range shows that small deviations in chemical composition or heat treatment

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from specified limits will readily change the microstructure, thereby producing effects contrary to those required.

As an illustration of this fact, consider the aluminum bronze having a composition of 9.5 per cent aluminum, balance copper. This alloy would be at the boundary of the single and 2-phase regions of the equilibrium diagram shown in Fig. 1. Alloys with aluminum contents lower than 9.5 per cent would consist, under

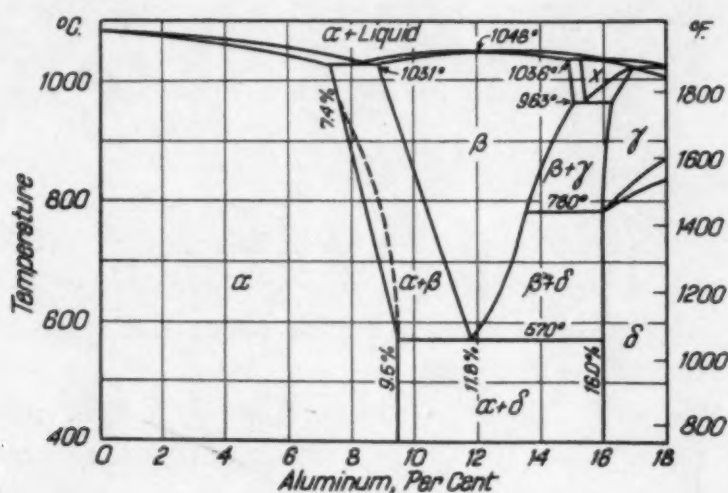


Fig. 1—Copper-Aluminum Constitution Diagram, METALS HANDBOOK, 1939 Edition, p. 1342.

equilibrium conditions, of only alpha solid solution; while those alloys with aluminum contents greater than 9.5 per cent but below 11.8 per cent, under equilibrium conditions, would consist of an eutectoid of alpha and delta solid solutions in a matrix of alpha. However, in commercial heat treatments, equilibrium conditions are not obtained, and usually a transformation product called "acicular" beta (1)¹ is retained at room temperature together with varying amounts of alpha and alpha plus delta eutectoid, depending on the cooling rate. Since the constituents "acicular" beta, alpha, and delta have different mechanical properties, the amounts and distribution of the constituents in the microstructure, as controlled by the chemical analysis and heat treatment, will determine to a great extent the ultimate properties of the alloy.

Among others, an important function of the metallographist is to determine from the microstructure of a specimen whether or not it has been properly heat treated to give the desired physical properties. To attain any degree of accuracy in studies of this kind, not only does

¹The figures appearing in parentheses pertain to the references appended to this paper.

the specimen have to be representative and well polished, but the etchant used must reveal the true structure of the metal.

HISTORY

The above fact became manifest in the course of an investigation of defective propeller parts made from aluminum bronze. The alloy investigated had a chemical composition in the following range:

Aluminum10.5 to 11.2 Per Cent
Iron 3.0 to 4.0 Per Cent
Others 0.4 Per Cent Maximum
CopperBalance

Metallographic examination of representative samples of these parts was obstructed by the unsatisfactory results obtained with the conventional etching reagents and the lack of metallographic data on alloys of the specified composition and grain size. Available literature on copper-aluminum alloys was investigated, but no information directly applicable to the specific problem was found. Consequently, it was decided to initiate a test program in which the microstructures of aluminum bronzes of the compositions given above could be correlated with various heat treatments and the resultant physical properties.

The effect of the iron in the alloys under question is a "negative replacement effect" on the aluminum content, in which 1 per cent iron will normally effect a 0.05 to 0.12 per cent downward displacement of aluminum (2). As a result of this effect, the exact temperature at which a homogeneous beta phase is produced on heating was not known. Therefore, this temperature and the proper cooling rate and draw temperature required to produce the desired physical properties had to be determined. In this manner, specifications eventually could be prepared which would enable control laboratories to inspect easily the heat treated product not only from the standpoint of hardness but of microstructure as well. Thus a "double check" on the alloy would be obtained, which is the usual procedure in metallurgical laboratories.

In such a specification, it was an absolute necessity to establish a metallographic etchant for aluminum bronze which would give reliable results and at the same time readily reproduce the same etched condition on a series of specimens. Moreover, the etchant had to be

easy to use for inexperienced metallographers, an important item considering the present man-power situation.

SPECIMEN PREPARATION

In the metallographic preparation of all the specimens used in this work, regardless of the etching technique, a well prepared, scratch-free surface was produced in the following manner:

1. Specimens were mounted in Bakelite.
2. Specimens were rough ground on a vertical wet grinder having a coarse and fine wheel with a speed of 1750 revolutions per minute. The two wheels were Norton's alundum Nos. 3860 J and 150 N, respectively.
3. Specimens were then fine ground on two lead lap wheels using American Optical Company's abrasive Nos. 302 and 303½, respectively, the latter of which produced the finer scratches. The speed of both wheels was 250 revolutions per minute.
4. Grinding was completed on canvas cloth to which a mixture of distilled water and 600-mesh carborundum was added. The speed of this wheel was also 250 revolutions per minute.
5. Preliminary polishing was conducted on a silk cloth with a speed of 1150 revolutions per minute, using chrome oxide as the polishing medium.
6. Final polishing was accomplished on "gamal" cloth (distributed by Eimer and Amend Company), with the polishing medium likewise chrome oxide. The speed of this wheel was 850 revolutions per minute.
7. Prior to etching, the aluminum bronze specimens were rinsed with alcohol and dried under an air blast.

ETCHANT TRIALS

The first attempt to etch specimens of aluminum bronze prepared by the method described above was to rub them with cotton soaked in a solution of the following composition:

NH ₄ OH (sp. g. 0.91)5 parts
H ₂ O ₂ (3 per cent)2 parts
H ₂ O5 parts

This etchant and its use are described in the METALS HANDBOOK (3). Microexamination at low magnification of a series of specimens etched in this manner revealed the gross differences in microstructure. However, good contrast between individual constituents at high magnifications was not obtained. An attempt to eliminate this con-

dition by repeated polishing and etching, as frequently used with steel, did not result in any success.

It was believed that rubbing the specimen during the etching process had blurred the etched surface. Consequently, the specimens were etched by dipping them in the aforementioned solution. As with the previously discussed method of etching, poor contrast was noted at high magnification which was not eliminated by repeated polishing and etching.

The results obtained with the two etching techniques indicated that for good contrast between individual constituents some etchant containing neither ammonium hydroxide nor hydrogen peroxide should be used. Therefore, an investigation was made of all the available literature distributed by manufacturers of copper-base alloys for another etchant which would yield satisfactory results. Aside from the ammonium hydroxide-hydrogen peroxide solutions, the most commonly used etchant appeared to be a ferric chloride-hydrochloric acid solution. This solution was also recommended in the METALS HANDBOOK (3) in various compositions. Following the techniques described in this reference, the authors obtained etching results on the aluminum bronze specimens which were considerably improved over those previously produced with solutions containing ammonium hydroxide and hydrogen peroxide. Nevertheless, desired results were not produced, and further investigation of the literature was deemed necessary.

A statement by Berglund (4) indicated that for an aluminum bronze alloy having a composition of 8.8 per cent aluminum, 3.7 per cent iron, and 87.5 per cent copper, good results were obtained with an etchant having the following composition:

HNO ₃ (sp. g. 1.42)	5 ml.
CrO ₃	20 g.
H ₂ O	75 ml.

This etchant was tried on the subject specimens and favorable results were obtained. However, it made visible scratches which otherwise were not apparent. Also the solution was nearly opaque and it was impossible to observe the etching reaction, thereby causing difficulty in reproducing the etch on a series of nonidentically heat treated specimens.

Two other etchants, a cold 10 per cent solution of ammonium persulphate suggested by Stockdale (5) and a saturated aqueous

solution of chromic acid suggested by Berglund (4), were then tried but gave very poor results. The first considerably overetched the specimens in 1 second, while the latter only slightly attacked the specimen after 2 minutes' immersion.

Proceeding on the basis of the fact that the etching of copper-base alloys is an oxidizing reaction, it was thought that an electrolytic etchant with an aqueous solution of chromic acid as the electrolyte would produce results comparable to those obtained with nitric acid plus chromic acid. Using a 20 per cent solution of chromic acid with a voltage of 6 volts D. C. and an aluminum sheet as the cathode (the aluminum bronze specimen was made the anode by touching it with a steel pointer), extreme overetching occurred in less than 1 second. The electrolyte was further diluted to 10 and 5 per cent chromic acid with the etching reaction becoming slower with each trial. Finally using a 1 per cent solution with the same voltage and cathode, excellent results were obtained. Very good contrast was observed between the constituents at low and high magnifications and a candid photomicrograph of the structure could be taken.

RESULTS

Figs. 2 through 15 are photomicrographs illustrating the action of the electrolytic etchant on various specimens of aluminum bronze. The etching time for these specimens using the same procedure described in the above paragraph varied from 3 to 6 seconds.

In all the aluminum bronze specimens investigated for this paper, no "acicular" beta as described and shown by Smith and Lindlief (1) was noted. It is believed that the addition of iron to the alloy increased the rate of transformation to the extent that this particular transformation product did not appear even with the most drastic quench used. This follows from the fact that a great part of the iron present would react with the aluminum at elevated temperatures to form the intermetallic compound, FeAl_3 , the presence of which would aid in the nucleation of the transformation products. No samples of aluminum bronze having "acicular" beta in the microstructure were available. However, it is believed with confidence that the electrolytic chromic acid reagent etchant would etch such a microstructure satisfactorily.

Smith and Lindlief (1) discuss the formation of another constituent which they call "beta prime" in an aluminum bronze con-

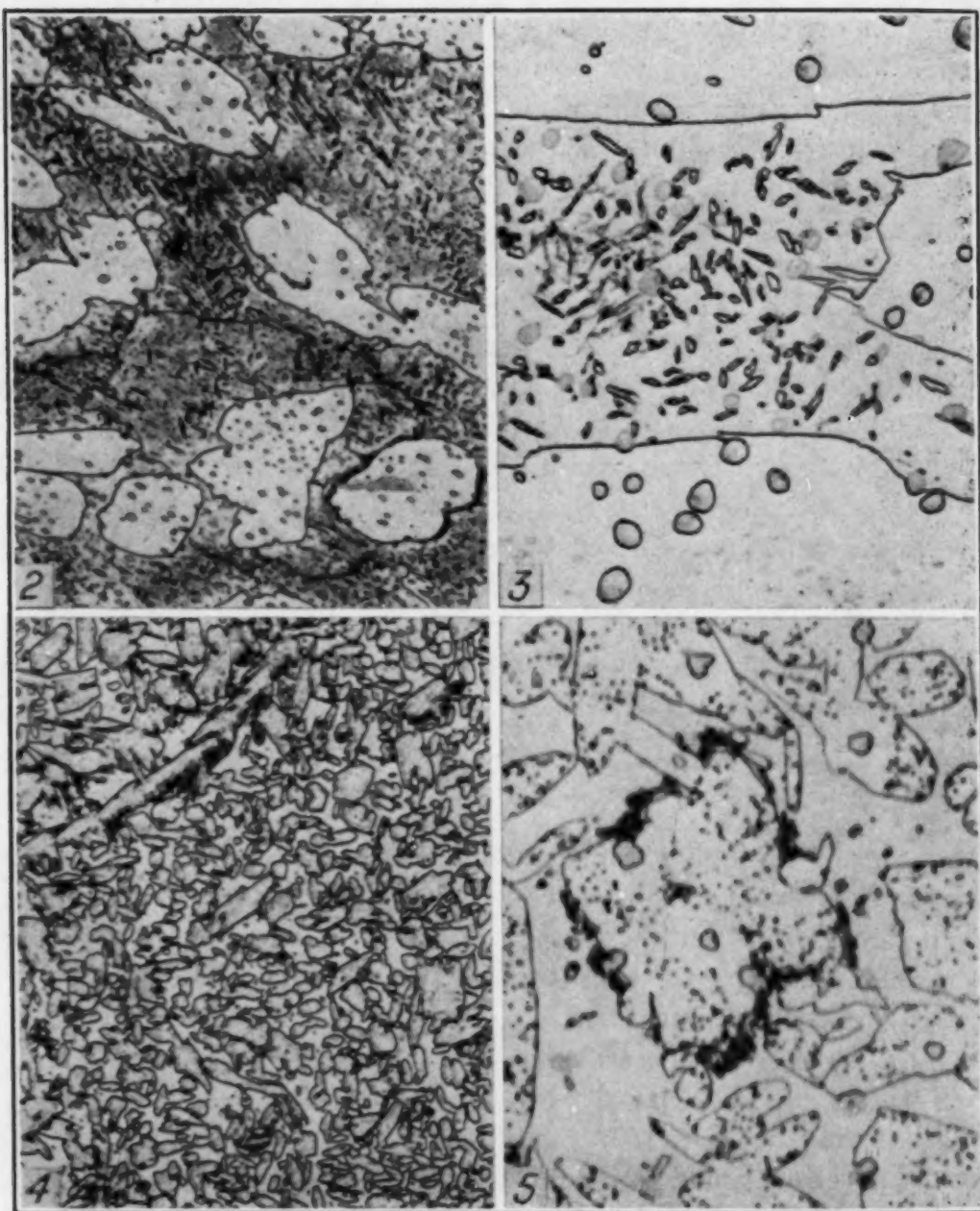


Fig. 2—Aluminum Bronze Containing 10.7 Per Cent Aluminum, 3.5 Per Cent Iron, Balance Copper. Furnace-Cooled from 1650 Degrees Fahr. (900 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Beta Partly Transformed to Alpha-Delta Eutectoid Plus Alpha Plus Gray Globules of FeAl_2 .

Fig. 3—Same as Fig. 2. $\times 1000$.

Fig. 4—Aluminum Bronze Containing 10 Per Cent Aluminum, 3.5 Per Cent Iron, Balance Copper. Air-Cooled from 1550 Degrees Fahr. (845 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Alpha Plus Small Amounts of Delta in Beta Matrix.

Fig. 5—Same as Fig. 4. $\times 1000$. Alpha and Alpha-Delta Eutectoid (Black Mottled) and FeAl_2 in Beta Matrix.

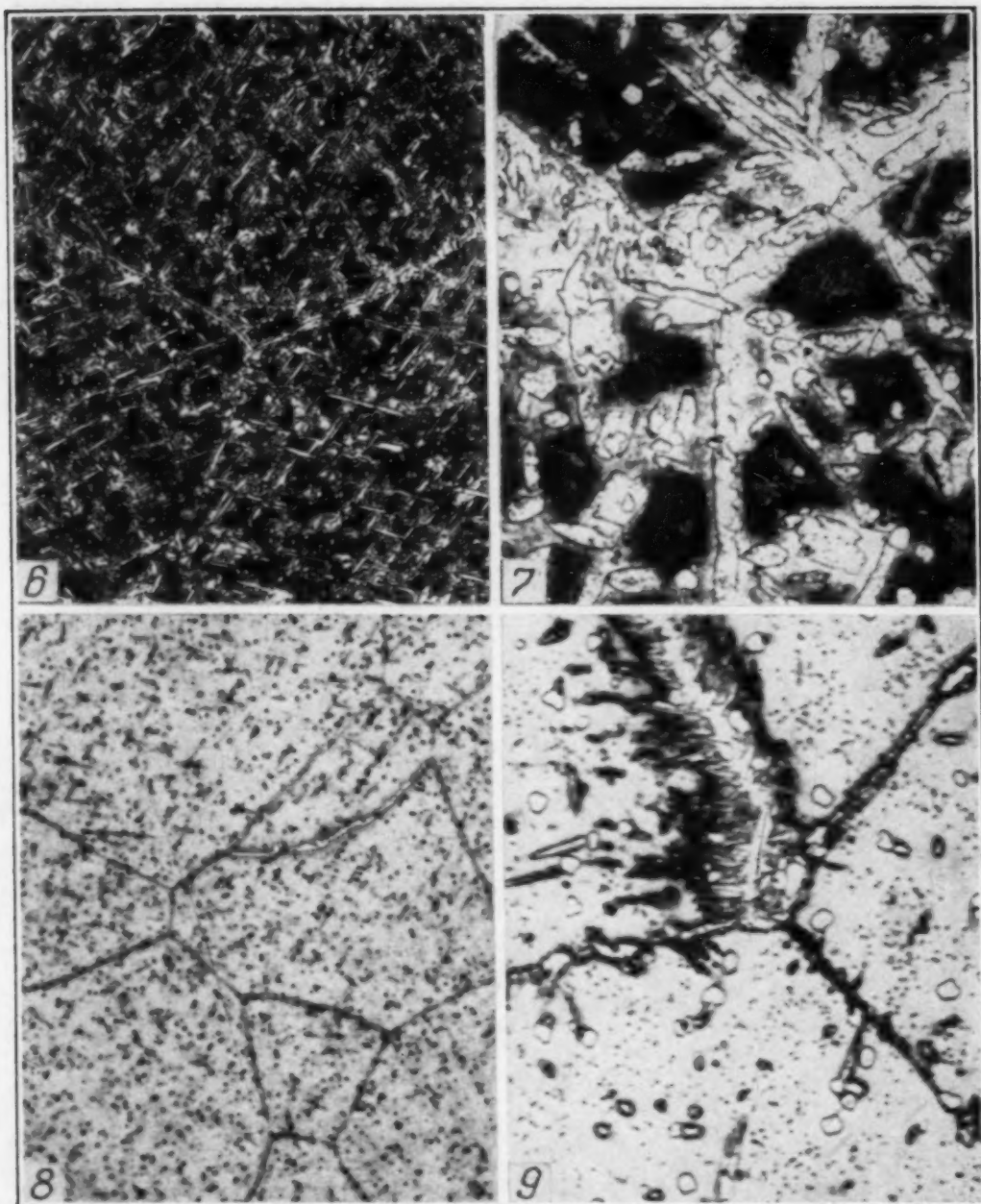


Fig. 6—Aluminum Bronze Containing 10 Per Cent Aluminum, 3.5 Per Cent Iron, Balance Copper. Oil-Quenched from 1550 Degrees Fahr. (845 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Alpha Needles (White) in Beta Matrix (Dark). Beta Etches Dark as a Result of Extremely Fine Dispersion of Alpha and Delta.

Fig. 7—Same as Fig. 6, Except Magnification is $\times 1000$. Note that Globules of $FeAl_3$ in this Case Appear White in the Dark Beta Matrix. White Needles are Alpha.

Fig. 8—Aluminum Bronze Containing 10 Per Cent Aluminum, 3.5 Per Cent Iron, Balance Copper. Water-Quenched from 1550 Degrees Fahr. (845 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Alpha (White) at Grain Boundaries and Along Crystallographic Planes in Beta (Gray) Matrix.

Fig. 9—Same as Fig. 8, Except Magnification is $\times 1000$. Alpha (White) at Grain Boundaries, $FeAl_3$ (White Globules) Plus Alpha-Delta Eutectoid in Beta Matrix.

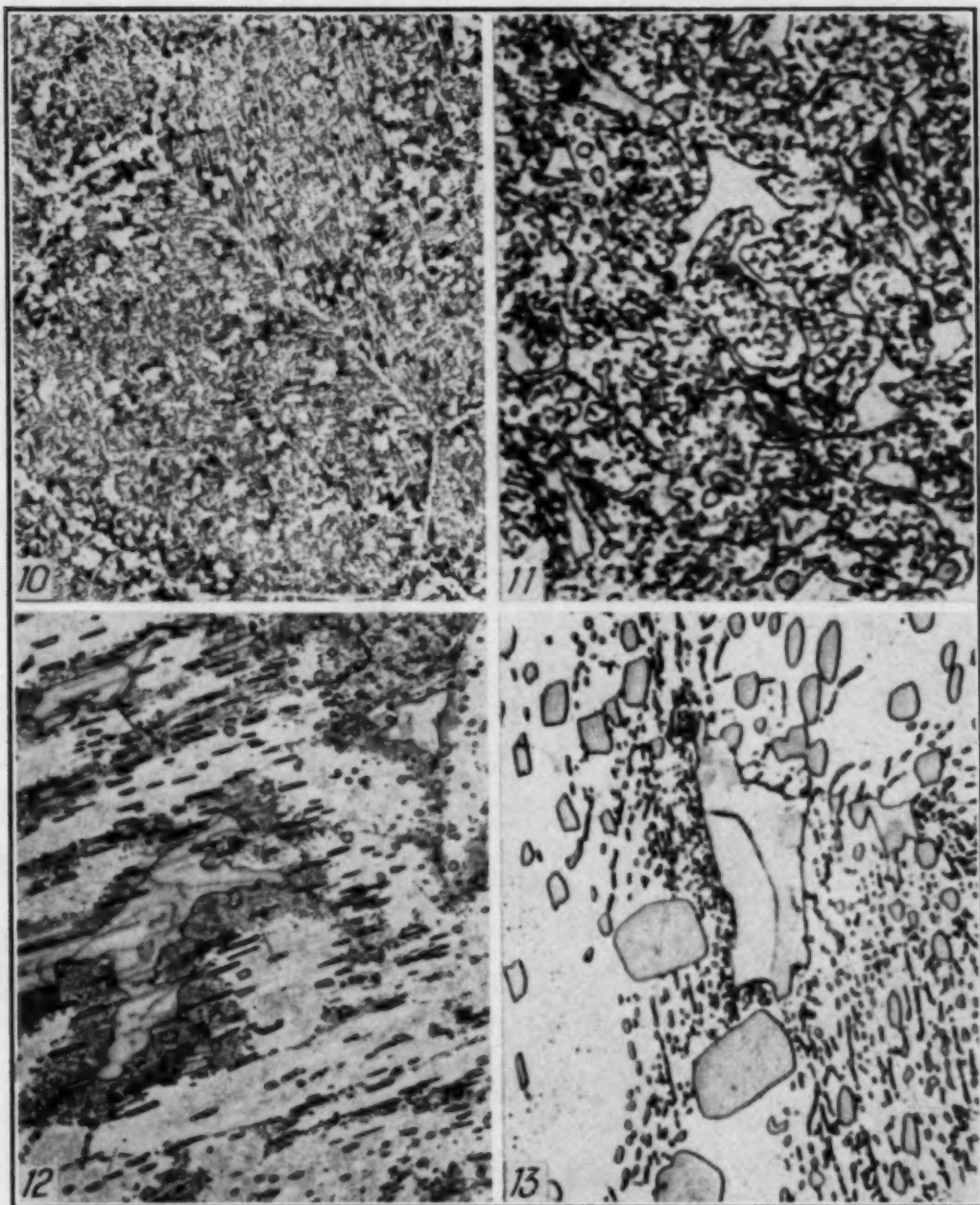


Fig. 10—Aluminum Bronze Containing 10 Per Cent Aluminum, 3.5 Per Cent Iron, Balance Copper. Quenched in Water from 1600 Degrees Fahr. (870 Degrees Cent.), and Drawn at 1170 Degrees Fahr. (630 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Alpha Plus Reprecipitated Alpha (Both White) and Delta in Beta Matrix.

Fig. 11—Same as Fig. 10 Except Magnification is $\times 1000$. Alpha (Primary and Secondary) Plus Alpha-Delta Eutectoid Plus FeAl_3 Globules in Beta Matrix.

Fig. 12—Aluminum Bronze Containing 10 Per Cent Aluminum, 3.0 Per Cent Iron, 5.0 Per Cent Nickel, 1.0 Per Cent Manganese, Balance Copper. Furnace-Cooled from 1650 Degrees Fahr. (900 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Alpha-Delta Eutectoid Plus $(\text{Fe:Ni})\text{Al}_3$ Plus Probably Supercooled Beta (Outlined in Center of Field) in Alpha Matrix.

Fig. 13—Same as Fig. 12. $\times 1000$.

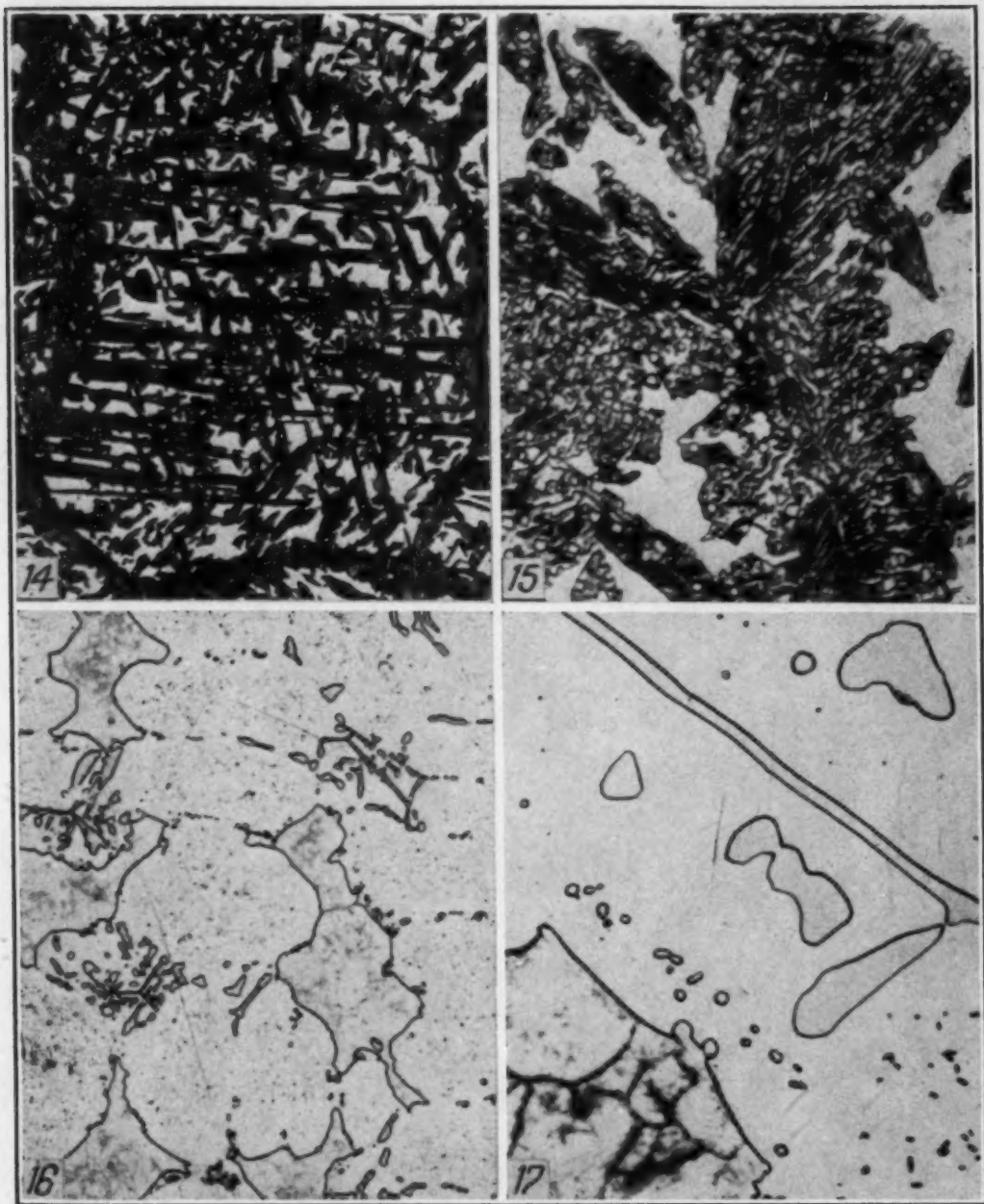


Fig. 14—Aluminum Bronze Containing 11.45 Per Cent Aluminum, 5.23 Per Cent Nickel, 4.7 Per Cent Iron, Balance Copper, Cooled in Air from 1650 Degrees Fahr. (900 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Alpha-Delta Eutectoid in Beta Matrix.

Fig. 15—Same as Fig. 14, Except Magnification is $\times 1000$. Note "Bainitic" Appearance of Alpha Plus Delta Eutectoid. Gray Globules are (Fe:Ni)Al₃.

Fig. 16—Copper-Beryllium Alloy Containing 2.60 Per Cent Beryllium, 1.20 Per Cent Nickel, and 96.20 Per Cent Copper. Quenched in Water from 1470 Degrees Fahr. (800 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Beta (Gray Outlined) and Nickel-Beryllium (Script) in Alpha Matrix.

Fig. 17—Same as Fig. 16 Except Magnification is $\times 1000$. Beta (Mottled Gray), Nickel-Beryllium (Clear Gray) and Gamma Spheroids in Alpha Matrix.

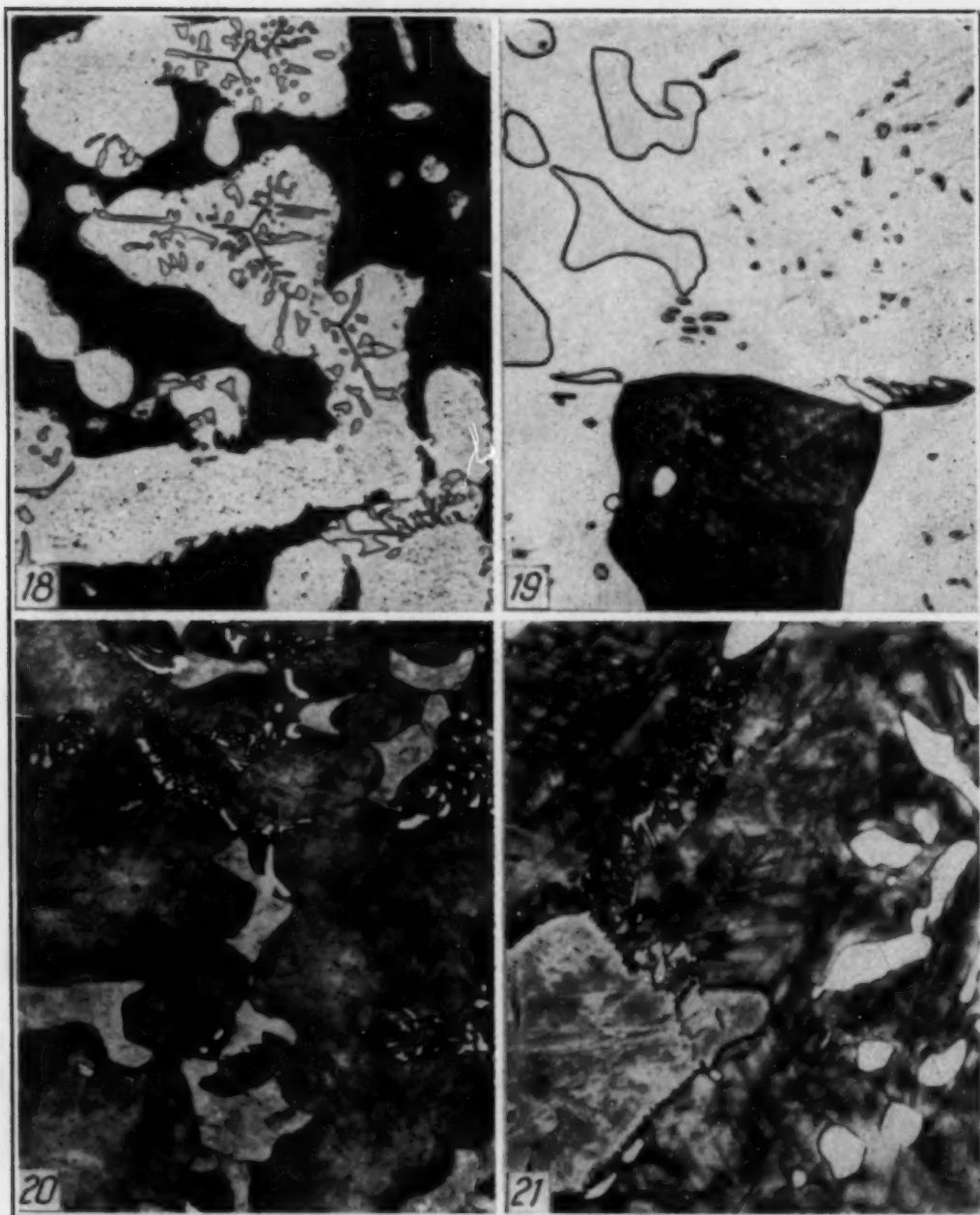


Fig. 18—Copper-Beryllium Alloy Containing 2.60 Per Cent Beryllium, 1.20 Per Cent Nickel, and 96.20 Per Cent Copper. Quenched in Water from 1470 Degrees Fahr. (800 Degrees Cent.) and Drawn $2\frac{1}{2}$ Hours at 570 Degrees Fahr. (300 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. Magnification: $\times 150$. Partially Transformed Beta (Dark), Nickel-Beryllium (Script) in Alpha (White).

Fig. 19—Same as Fig. 18 Except Magnification is $\times 1000$. Partially Transformed Beta (Dark), Nickel-Beryllium (Outlined Gray) and Gamma Spheroids in Alpha Matrix. Note Beginning Precipitation of Gamma in Alpha.

Fig. 20—Copper-Beryllium Alloy Containing 2.60 Per Cent Beryllium, 1.20 Per Cent Nickel, and 96.20 Per Cent Copper. Quenched in Water from 1470 Degrees Fahr. (800 Degrees Cent.), and Drawn 3 Hours at 570 Degrees Fahr. (300 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Partially Transformed Beta (Grey Outlined) and Nickel-Beryllium (White Script) and Gamma Spheroids in Alpha Matrix. Note Beginning Precipitation of Gamma in Alpha.

Fig. 21—Same as Fig. 20 Except Magnification is $\times 1000$. Partially Transformed Beta (Grey) and Nickel-Beryllium (White Script) in Precipitation Hardened Alpha. Note Gamma Particles at Grain Boundaries.

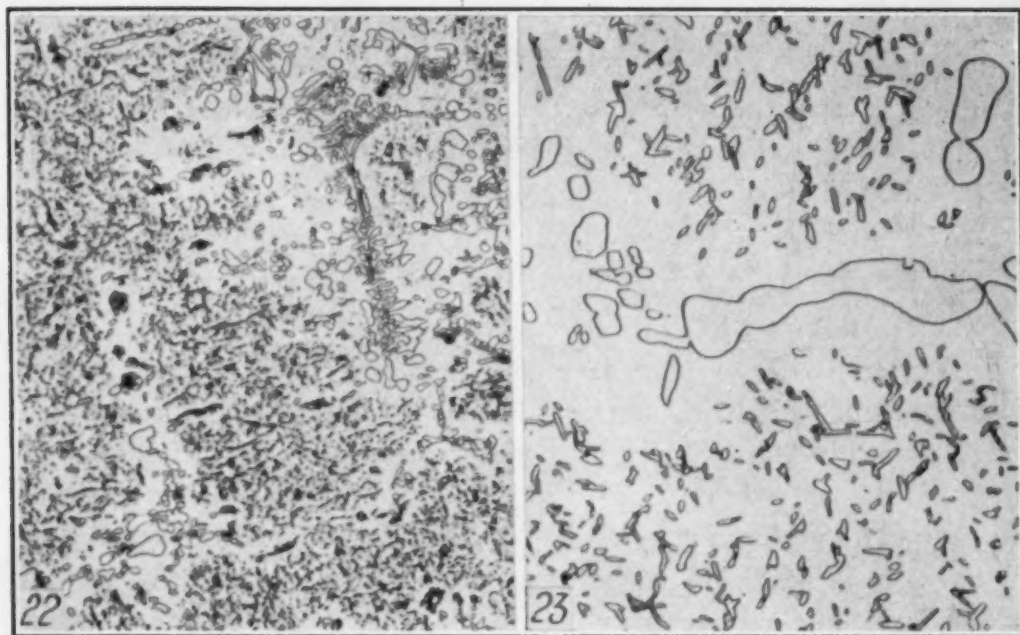


Fig. 22—Copper-Beryllium Alloy Containing 2.60 Per Cent Beryllium, 1.20 Per Cent Nickel, 96.20 Per Cent Copper. Quenched in Water from 1470 Degrees Fahr. (800 Degrees Cent.), and Drawn for 5½ Hours at 570 Degrees Fahr. (300 Degrees Cent.). Etched Electrolytically in 1 Per Cent Chromic Acid. $\times 150$. Precipitation Complete: Gamma Specks and Nickel-Beryllium in Alpha Matrix.

Fig. 23—Same as Fig. 22 Except Magnification is $\times 1000$. Elongated Particles of Gamma and Nickel-Beryllium (Gray Outlined) in Alpha Matrix.

taining 11.87 per cent aluminum; 0.06 per cent iron; 0.02 per cent silicon; 0.01 per cent tin; balance copper. This constituent appeared in the form of rosette shaped particles only on etching with either a solution of 50 per cent 0.90 sp. gr. ammonium hydroxide plus 10 per cent of 9 per cent hydrogen peroxide or the following solution:

HNO ₃ (1.42 sp. gr.)40 grams
CrO ₃25 grams
H ₂ O35 grams

They found that "beta prime" appeared in specimens isothermally transformed at 535 degrees Cent. (995 degrees Fahr.) or lower, and in specimens continuously cooled at rates more rapid than 192 degrees Cent. per hour.

At first glance the "beta prime" constituent appears to be present as spheroidal particles in the photomicrographs of Figs. 2 to 15. However, there was ample proof that these spheroids were not the beta prime constituent, but instead the intermetallic compound FeAl₃, or a complex (Fe:Ni)Al₃. First, all of the spheroids were seen on the unetched specimens and subsequent etching had no effect on them. Second, while a few of the particles were rosette shaped, the

majority were globular. Third, all of the particles had a faint bluish-gray tinge, both in the etched and unetched condition. Fourth, etching the specimens in a 0.5 per cent hydrofluoric acid solution blackened the particles, thereby logically establishing them as FeAl_3 or $(\text{Fe:Ni})\text{Al}_3$ (6).

USE OF THE ETCHANT FOR COPPER-BERYLLIUM ALLOYS

As mentioned previously in this paper, all etchants for copper-base alloys are oxidizing agents. Since the 1 per cent chromic acid reagent used electrolytically falls under this category, it was thought that this etchant with modifications might be successful with other alloys than aluminum bronze. The only copper base alloys which were available to the authors at the time of this writing were various heat treated specimens of copper-beryllium having the following composition:

Beryllium	2.60 Per Cent
Nickel	1.20 Per Cent
Copper	96.20 Per Cent

Results of using the electrolytic 1 per cent chromic acid etchant for 5 seconds on the copper-beryllium were as successful as those obtained on the aluminum bronze specimens. Photomicrographs and data relating to the heat treatment and microstructure of the copper-beryllium alloy are given in Figs. 16 to 23, inclusive. Note that the etchant clearly revealed the transformation which occurred for different age hardening periods.

CONCLUSIONS

The photomicrographs selected for this paper are presented as evidence that 1 per cent chromic acid used electrolytically as an etchant for aluminum bronze and copper-beryllium is superior to those generally recommended for alloys of this type. Its advantages lie in its simplicity and effectiveness. Moreover, the etch is quickly and surely reproducible on a series of specimens. Care must be taken, however, to use distilled water for the solvent as the common salts found in tap water produce undesired staining.

Time was not available for additional trials of the etchant on other copper-base alloys. Nevertheless, they are aware of no factors

which would prevent results with other copper-base alloys comparable to those obtained with aluminum bronze and copper-beryllium.

ACKNOWLEDGMENT

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THE MECHANISM OF FAILURE OF 18 CR-8 NI CRACKING STILL TUBES

BY C. L. CLARK AND J. W. FREEMAN

Abstract

This paper presents results obtained from a metallurgical examination of 18 Cr-8 Ni cracking still tubes which had been in service for time periods ranging up to 97,520 hours. In the "as-service" condition these tubes possessed entirely different characteristics for (a) some were still ductile on the basis of the flattening test, (b) others were brittle but could have their ductility restored, while (c) others were permanently brittle.

On the basis of the results obtained, it is believed that the deterioration and possible actual failure of 18 Cr-8 Ni cracking still tubes in service is due to structural changes at the grain boundaries which are progressive in nature and are dependent on time, temperature and stress. These structural changes consist first of the precipitation and growth of highly alloyed ferrite due to the decomposition of austenite. When these precipitated areas have grown to a certain size, cracking, at first nearly submicroscopic in nature, will occur under certain conditions of time, temperature and stress. When this cracking has occurred the tube is permanently brittle but at any time prior to the cracking the tube is either ductile or can have its ductility restored.

THE austenitic 18 Cr-8 Ni steel has found wide use in the petroleum industry during the past 10 to 15 years as cracking still tubes in those units where the corrosion has been very severe, due to the processing of the so-called sour crudes. In general the performance of this steel has been satisfactory and many of the original tubes are still in service after operating time periods up to 100,000 hours (11.4 years). In general, the temperature of the tubes in these installations has been held to a maximum of 1200 to 1250 degrees Fahr. (650 to 675 degrees Cent.).

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However, during this time period, certain of these tubes have actually failed while others have undergone rather pronounced changes in their physical properties. As a result of these experiences with 18 Cr-8 Ni tubes, the following questions can be raised:

1. Is the failure caused by progressive intergranular oxidation, or corrosion, either at the normal operating temperature of 1200 to 1250 degrees Fahr. (650 to 675 degrees Cent.), or during possible relatively short periods of overheating at the more elevated temperatures?

2. Or is this failure primarily due to structural changes which occur within the steel either at normal operating temperatures or during overheating? If structural change is responsible, what is its character?

3. Why can the ductility of embrittled tubes, as measured by the flattening test, sometimes be restored by heating to 1500 to 1900 degrees Fahr. (815 to 1040 degrees Cent.) while, at other times, this treatment is ineffective?

As a result of new processes, introduced by the present emergency, 18 Cr-8 Ni tubes are now operating at metal temperatures up to the order of 1650 degrees Fahr. (900 degrees Cent.). This has introduced other interesting metallurgical problems but the present paper is confined to those cases in which the maximum normal operating temperature has been 1200 to 1250 degrees Fahr. (650 to 675 degrees Cent.).

MATERIALS INVESTIGATED

Through the co-operation of certain petroleum companies, cracking still tubes of 18 Cr-8 Ni were obtained which had been in service for time periods ranging from 35,000 to 97,500 hours. Certain of these tubes had actually failed in service, some were still ductile, others were brittle but could be rejuvenated, while still others were permanently embrittled. Pertinent data with respect to these tubes are summarized in Table I.

Insofar as the chemical composition is concerned, all of the analyses, except that of Tube A, are within the present range for 18-8, although there are certain variations in the carbon, chromium and nickel contents. It is worthy of note that Tube A, whose carbon content is slightly beyond the 0.08 per cent maximum now permitted, became brittle during service but it could have its ductility

restored by rejuvenation treatments at 1500 to 1900 degrees Fahr. (815 to 1040 degrees Cent.). On the other hand, Tube C-2, containing lower carbon, became permanently brittle during service.

Likewise, all the tubes were coarse-grained, the A.S.T.M. grain size being 4 or coarser. These tubes were, of course, processed sev-

Table I
Pertinent Data on 18-8 Cracking Still Tubes Investigated

<i>A. Chemical Composition</i>											
Tube Designation	C	Mn	P	S	Si	Cr	Ni	Cu	N ₂	Al	Sn
A	0.085	0.44	0.015	0.014	0.74	17.37	9.13	0.12	0.042	0.010	0.01
B	0.07	0.42	0.015	0.011	0.33	18.28	9.13	0.11	0.103	0.005	0.02
C-1	0.07	0.43	0.014	0.012	0.44	17.63	9.07	0.07	0.040	0.008	0.01
C-2	0.07	0.42	0.015	0.011	0.41	17.55	9.11	0.05	0.040	0.005	0.01
D	0.06	0.36	0.017	0.013	0.34	18.13	8.54
E	0.06	0.34	0.017	0.014	0.36	18.01	8.44

<i>B. Service Life and Reported Condition After Service</i>		
Tube Designation	Service Life Hours	Condition After Service
A	35,000	One section cracked. Balance of tube brittle but capable of rejuvenation at 1500 or 1900 degrees Fahr. (815 or 1040 degrees Cent.).
B	40,055	One section cracked. Balance of tube brittle with erratic response to rejuvenation treatment.
C-1	78,000	Section of tube still ductile after service.
C-2	78,000	This section of tube is opposite end of Tube C-1. Certain sections cracked in service and balance was permanently brittle.
D	97,500	Fairly ductile after service and capable of rejuvenation.
E	97,520	Fairly ductile after service and capable of rejuvenation.

NOTE: Comparative ductility determined by flattening tests on complete tube rings cut from the respective tubes.

eral years ago and at that time it was general practice to water-quench or air-cool from temperatures of 1900 degrees Fahr. (1040 degrees Cent.) or higher, and either of these treatments produces a coarse grain size in this steel.

INVESTIGATIONAL PROCEDURE

In addition to the chemical analyses, given in Table I, all of the tubes were submitted to a visual and macroscopic examination, to flattening tests, to a magnetic examination, to the Strauss corrosion tests and to a microscopic examination. In addition, a hardness survey, room temperature tensile and 1200-degree Fahr. (650-degree Cent.) stress-rupture tests were conducted on those tubes for which there was sufficient material available.

EXPERIMENTAL RESULTS

The results obtained from the various tests undertaken are summarized in the following sections:

Visual Examination—Differences existed in the physical appearance of the various tube sections. Tubes A, C-1 and C-2 showed slight general oxidation with the scale layer being thin and tightly adherent, while there was little evidence of oxidation on Tubes D and E. Tube B showed a condition on the outside surface, often referred to as fish scale, and probably resulting from intergranular attack.

Only one of the tube sections submitted, Tube B, was cracked to failure and this crack occurred on the fire side. The sections of Tubes A, C-1 and C-2, were taken from tubes which had cracked in service but the sections submitted were from positions an appreciable distance from the actual failure. Tubes D and E had not failed in service but were removed because the wall thickness had decreased to the limit set by the refinery.

Measurements of the tube wall showed some thinning on the fire side and an increase in the diameter across this point, thus indicating that plastic deformation, or creep, had occurred during service. However, the decrease in wall thickness, and increase in diameter, were both of a low order of magnitude. Some of the tubes likewise showed evidence of metal loss on the inner surface due to corrosion and the action of the tube cleaner. Purely on the basis of visual inspection all of the tube section submitted, except Tube B which was actually cracked, would have been rated as capable of further service in cracking units.

Macro-Examination—The macrostructures of complete rings taken from each of the tube sections are shown in Fig. 1. Although only one ring from each tube is shown in this figure, several were taken at different points along each tube and the comments given are based on this complete examination. The macrostructures of all the tube sections, with the possible exception of Tube B, showed sound metal free from visual defects. In the case of Tube B small circumferential bursts are visible in the tube wall but these are believed to have occurred in service rather than having been present in the original tube.

While all of the tubes were of good quality the following differences were noted in their etching characteristics:

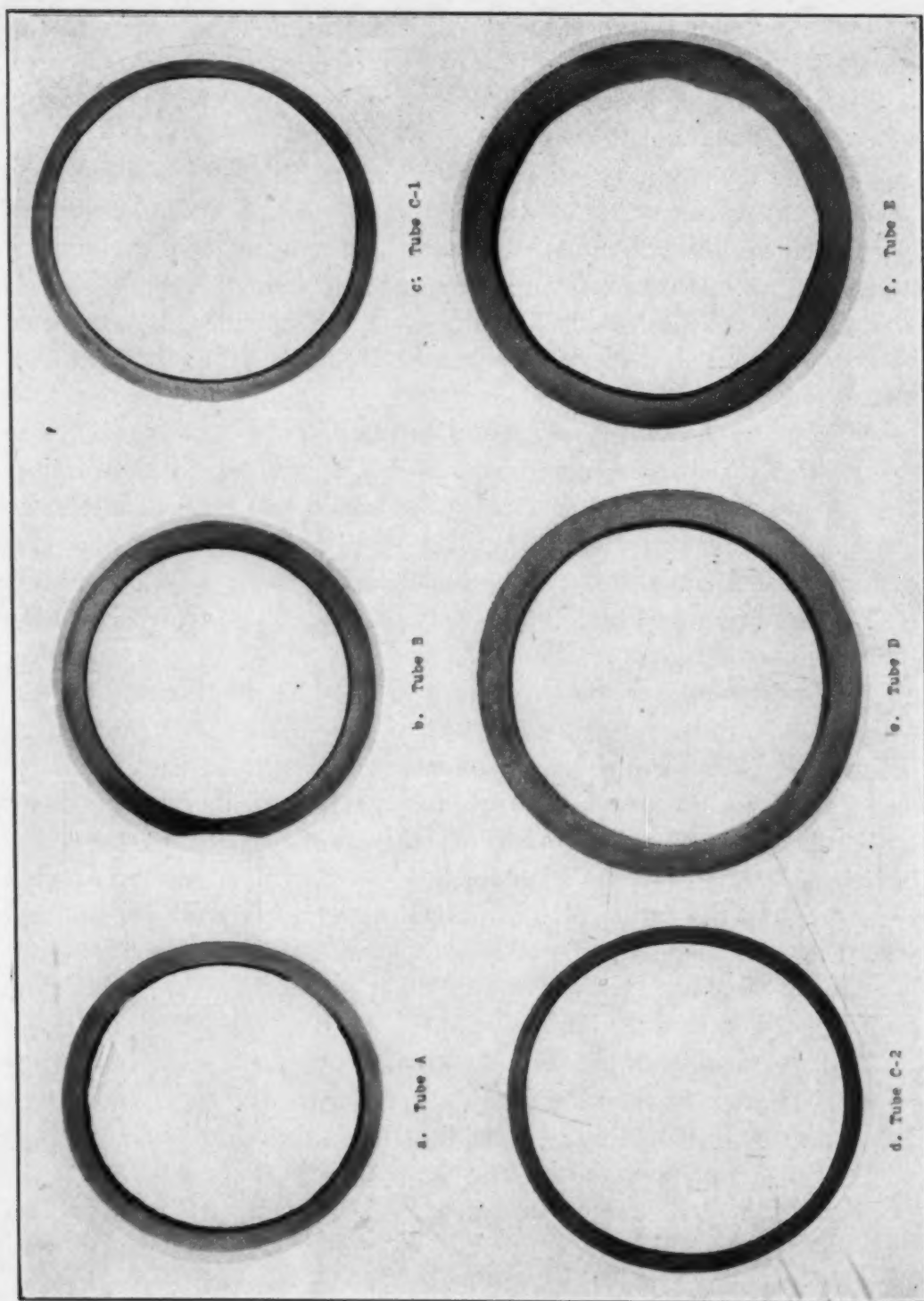


Fig. 1—Macrostructures of 18-8 Tubes After Service.

1. One section of the circumference of Tubes A, B, D and E etched more readily and deeper than the remaining areas, with the more readily attacked region corresponding to the fire-side during service. The width of this deeper etched area was more extended in Tubes A and B.

2. Tubes C-1 and C-2 etched uniformly around the entire circumference but the tubes differed in that the permanently brittle Tube C-2 etched more readily and deeper, being similar in this respect to the fire side of the remaining tubes.

3. In almost every case a narrow band on the inner surface etched to a greater extent than the balance of the tube wall. As will be shown later this condition is due to slight carburization and to cold working produced by the tube cleaners.

In general, it may be concluded that while the macro-examination revealed differences in structure it did not indicate the cause of brittleness in certain of the 18-8 tube sections after service.

Flattening Characteristics—The flattening test is the means often employed to determine the ductility characteristics of tubes after service. It has been found to be much more selective in this respect than are the ductility values obtained from the tensile tests. Accordingly, a large number of rings from each of the tube sections were subjected to this test both in the "as-service" condition as well as after the so-called rejuvenation treatments at 1500 and 1900 degrees Fahr. (815 and 1040 degrees Cent.). In addition, tube rings were flattened after certain amounts had been machined from the inner and outside walls in order to ascertain if surface attack might be a contributing cause, at least, of the brittleness. The location of each of the test rings, the manner in which they were treated, and the results obtained from the flattening tests are shown diagrammatically in Fig. 2. The following comments may be made with respect to the flattening characteristics of each of the tubes:

1. Tube A was brittle in the "as-service" condition but its ductility could be fully restored by a ½-hour heating period at 1500 or 1900 degrees Fahr. (815 or 1040 degrees Cent.). On the other hand, machining 0.050 inch from either the outside or inside surface had little if any influence on the degree of flattening.

2. Tube B was likewise brittle in the "as-service" condition but it exhibited erratic response to rejuvenation at 1500 and 1900 degrees Fahr. (815 and 1040 degrees Cent.).

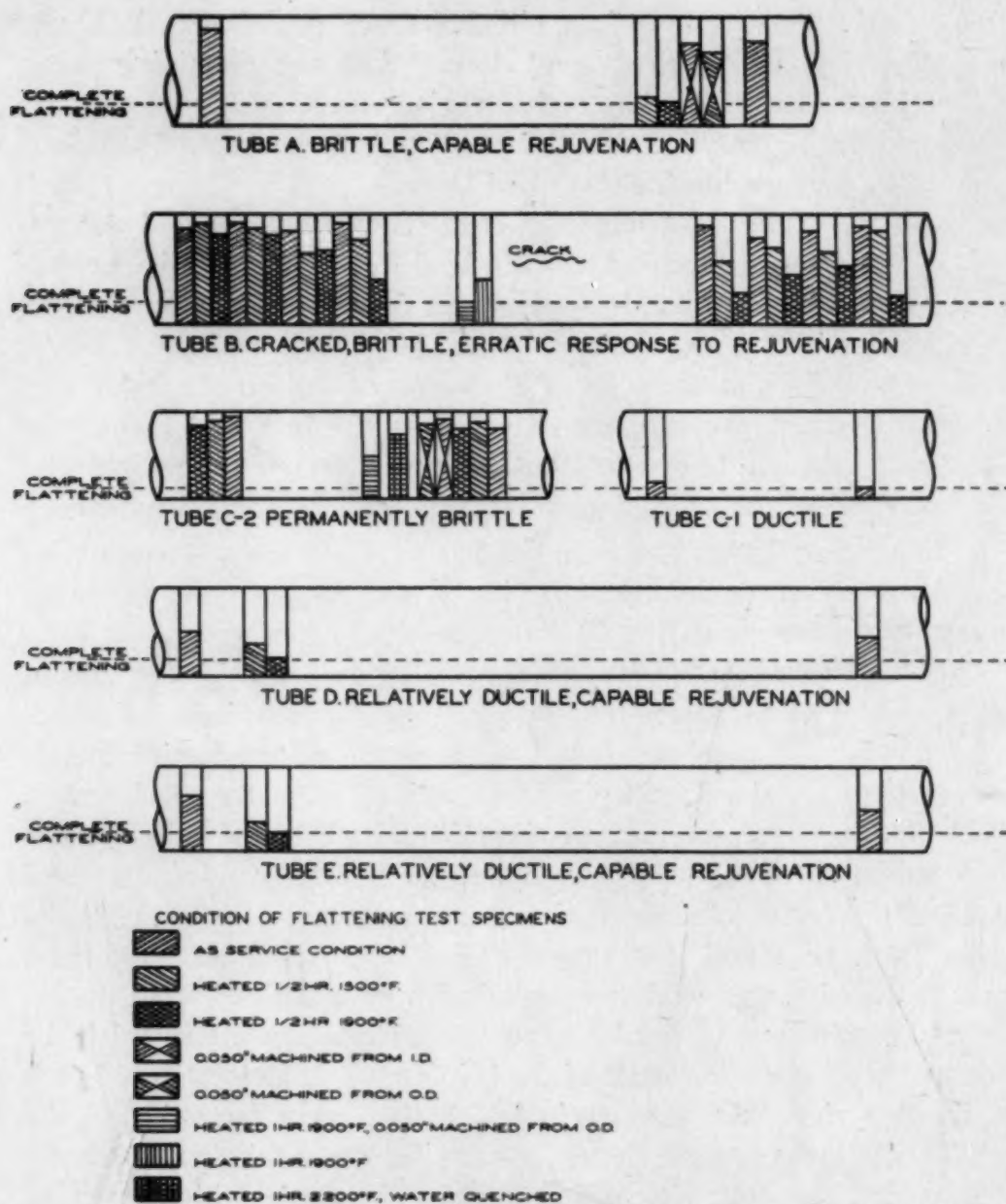


Fig. 2—Results of Flattening Tests on Five 18-8 Chromium-Nickel Tubes After Designated Heat Treatments.

3. Tube C-2 was classified as permanently brittle for complete ductility could not be restored by any of the treatments used. This was true even when an annealing temperature of 2200 degrees Fahr. (1205 degrees Cent.), followed by water quenching, was used. Likewise machining metal from either the outside or inside wall did not improve the flattening properties.

4. On the other hand, Tube C-1, which was from the same tube length as Tube C-2, but from the opposite end, was completely ductile in the "as-service" condition.

5. Both Tubes D and E possessed a good degree of ductility in the "as-service" condition. In both cases the ductility was further improved by the 1500-degree Fahr. (815-degree Cent.) treatment but the tube rings could only be completely flattened after the 1900-degree Fahr. (1040-degree Cent.) heat treatment.

Table II
Room Temperature Tensile Properties of Three 18 Cr-8 Ni Tubes
After Prolonged Service

Tube Designation	Service Life Hours	Location of Specimen	Tensile Strength Lb./Sq.In.	Yield Point Lb./Sq.In.	Elongation in 2 In. Per Cent
A	35,000	Fire Side	92,500	32,600	38.0
		Opposite Fire Side	96,700	33,250	41.0
		90° to Fire Side	96,800	25,700	41.5
		90° to Fire Side	95,800	26,500	44.0
D	97,500	Fire Side	84,300	29,000	30.5
		Opposite Fire Side	86,400	32,000	30.5
		90° to Fire Side	89,100	35,000	47.0
		90° to Fire Side	87,100	34,000	40.5
E	97,520	Fire Side	95,400	21,600	39.5
		Opposite Fire Side	99,400	24,800	44.0
		90° to Fire Side	96,500	21,000	45.5
		90° to Fire Side	96,500	29,900	44.5

NOTE: Yield point obtained by dividers. Strip specimens 1 inch wide at gage length.

In addition to the above observed differences in the overall flattening characteristics of these tubes, differences likewise existed with respect to the location of the fracture. In all of the tubes except C-2, the fracture, when it did occur, was generally confined to the fire side of the tube, while with Tube C-2 it was not.

On the basis of these findings, it is evident that when the flattening test is used to evaluate the ductility characteristics of tubes after service, pronounced differences may be found to exist not only in different tubes but in various sections of the same tube as well as around the circumference of a given tube ring.

Room Temperature Tensile Properties—Because of the amount of stock available tensile tests could only be conducted on Tubes A, D and E. These tubes were considered only in the "as-service" condition and strip tensile specimens were taken at the fire side, opposite the fire side, and 90 degrees to the fire side. The results obtained are given in Table II.

The variations found in the properties within each tube were not very pronounced, the only consistent difference being a somewhat lower strength and ductility on the fire side. The strength values of Tubes A and E were somewhat higher than those generally obtained in quenched 18 Cr-8 Ni tubes but this increase may be due either to the cold working on the inner surface, caused by the tube cleaner, or to structural changes.

The outstanding finding from these tests is that all of the tubes showed reasonably good ductility in the "as-service" condition while

Table III
Hardness Survey of Rings from 18 Cr-8 Ni Tubes After Prolonged Service

Tube Designation	Service Life Hours	Location of Specimen	Rockwell "B" from Outside (to Inside of Tube)
A	35,000	Fire Side	67, 81, 82, 82, 82, 82
		Opposite Fire Side	75, 81, 81, 82, 85, 84
		90° to Fire Side	78, 82, 82, 82, 83, 84
		90° to Fire Side	73, 82, 82, 83, 85, 84
D	97,500	Fire Side	77, 83, 85, 85, 89, 90
		Opposite Fire Side	82, 85, 85, 85, 86, 93
		90° to Fire Side	84, 85, 86, 86, 86, 88
		90° to Fire Side	85, 86, 86, 86, 88, 90
E	97,520	Fire Side	71, 79, 80, 79, 81, 80
		Opposite Fire Side	64, 78, 78, 79, 79, 80
		90° to Fire Side	68, 77, 78, 79, 80, 81
		90° to Fire Side	63, 77, 78, 79, 81, 80

the flattening tests showed Tube A to be brittle when in this condition. In other words, the flattening test is more selective and critical for this purpose than the tensile test.

Hardness Survey—A hardness survey was made around the circumference and across the wall of tube rings from each of the tubes. Typical results for Tubes A, D and E are given in Table III. The three tubes differed in hardness, that for Tube A ranging from 67 to 85, that of Tube D from 77 to 93, and that of Tube E from 63 to 81 Rockwell "B". Likewise as shown by the preceding values there were hardness differences within each tube. In general, the hardness tended to be harder towards the inner surface and this increase is due to a slight degree of carburization as well as to the cold working during tube cleaning.

Strauss Corrosion Resistance—The Strauss corrosion test was conducted on 1/8-inch square rings machined from the center of the tube wall of each of the tube sections. These tests were conducted in the "as-service" condition as well as after rejuvenation treatments at 1500 and 1900 degrees Fahr. (815 and 1040 degrees Cent.). Like-

wise certain of the specimens were heated at 1100 and 1200 degrees Fahr. (595 and 650 degrees Cent.) prior to the test. The results obtained are summarized in Table IV. The following conclusions are indicated by these findings:

1. All of the tubes were susceptible to the Strauss corrosion test in the "as-service" condition and all were immune after the rejuvenation treatments at either 1500 or 1900 degrees Fahr. (815 or 1040 degrees Cent.).

Table IV
Results from Strauss Corrosion Tests on Ring Samples of 18 Cr-8 Ni Still Tubes
After Prolonged Service

Tube Designation	Service Life Hours	Condition	Time for Failure Hours	Type Failure	Location of Failure
A	35,000	As Service	30	General	Every Place but Fire Side
		1500° F.—1 Hr.	N.F.	—	—
		1900° F.—1 Hr.	N.F.	—	—
B	40,055	As Service	23	General	Every Place but Fire Side
		1500° F.—1 Hr.	N.F.	—	—
		1900° F.—1 Hr.	N.F.	—	—
C-1	78,000	As Service	195	General	Complete Ring.
		1500° F.—1 Hr.	N.F.	—	—
		1900° F.—1 Hr.	N.F.	—	—
		1100° F.—24 Hr.	386	Localized	45° from Fire Side
C-2	78,000	1200° F.—24 Hr.	386	Localized	45° from Fire Side
		As Service	150	Localized	Single Point Attack
		1500° F.—1 Hr.	N.F.	—	—
		1900° F.—1 Hr.	N.F.	—	—
		1100° F.—24 Hr.	288	General	Complete Ring
D	97,500	1200° F.—24 Hr.	288	General	Complete Ring
		As Service	528	Localized	At Fire Side
		1500° F.—1 Hr.	N.F.	—	—
E	97,520	1900° F.—1 Hr.	N.F.	—	—
		As Service	45	Localized	90° from Fire Side
		1500° F.—1 Hr.	N.F.	—	—
		1900° F.—1 Hr.	N.F.	—	—

N.F. = Not Fractured in 528 hours.

2. However, differences did exist in the "as-service" specimens in the location of the attack around the circumference of the rings. Tubes A and B were attacked at all locations except at the fire side, with Tubes C-2 and D the attack occurred only at the fire side, with Tube C-1 the attack was general while with Tube E it was confined to about 90 degrees from the fire side.

3. Heating the "as-service" tubes at 1100 or 1200 degrees Fahr. (595 or 650 degrees Cent.) increased the time required for, and changed the location of, the attack.

These findings would, therefore, indicate that the Strauss corrosion results do not differentiate between these tubes in the same manner as the flattening tests.

Magnetic Characteristics—A qualitative measure of the comparative magnetic characteristics of the different tubes was obtained by measuring the angle through which specimens suspended by a thread could be pulled with a strong permanent magnet. It is recognized that this procedure is not too accurate but it is sufficiently sensitive to reveal rather slight differences in the magnetic characteristics of austenitic steels provided all test details are held constant.

Significant differences were revealed in the magnetic characteristics of specimens from each of these tubes. Specimens from the fire side of Tubes A and B responded to the magnet five times as strongly as specimens from the remainder of the tube walls. All portions of the circumference of Tube C-2 were as magnetic as the fire side sections of Tubes A and B while Tube C-1 was even less magnetic than the specimens away from the fire side of Tubes A and B. Tubes D and E were slightly more magnetic than Tube C-1, with Tube D being the more magnetic of these two.

The results from the magnetic tests on the "as-service" tubes are therefore in fairly good agreement with the flattening tests as the most brittle tube had the greatest magnetism and the most ductile tube the least. Likewise, the position of maximum magnetism around the circumference coincided with the location of cracking in the flattening test. On the other hand, the rejuvenation treatment at 1500 degrees Fahr. (815 degrees Cent.) removed little if any of the magnetism while it was, in many cases, beneficial in improving the flattening characteristics. The treatment at 1900 degrees Fahr. (1040 degrees Cent.) did, however, completely remove the magnetism.

Rupture Properties at 1200 Degrees Fahr. (650 Degrees Cent.)—Because of the amount of material available the rupture tests were conducted on a limited number of specimens from Tubes A, D and E and confined to a temperature of 1200 degrees Fahr. (650 degrees Cent.). Because of the previously noted differences in physical properties around the circumference of certain of these tubes, specimens were taken at the fire side, opposite the fire side and 90 degrees to the fire side. The dimensions of the tube wall required the use of 0.250-inch diameter by 1-inch gage length specimens. All tests were confined to the "as-service" condition. The results obtained from these tests, given in Table V and Fig. 3, indicate the following:

1. In each tube the fire side was considerably weaker than the other three sides. However, only in the case of Tube D did this section have low ductility.

- 2. In each tube the material opposite the fire side had the highest strength in the rupture test.
- 3. Tube A, which was classified as "brittle" by flattening tests on the "as-service" tube, had higher rupture strength and ductility than the other two tubes or water-quenched bar stock. Tube D possessed the lowest strength.

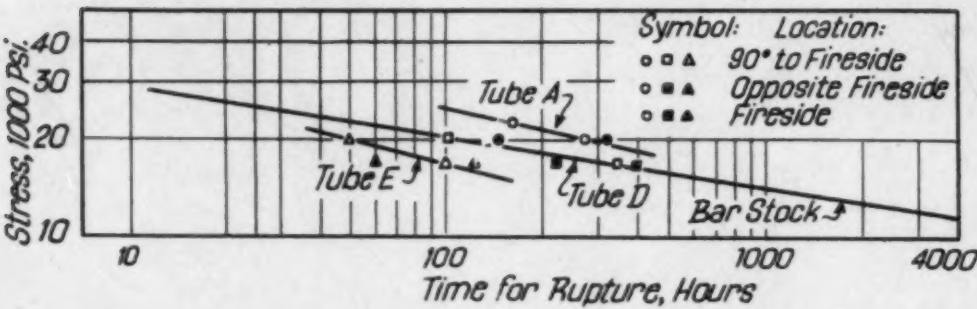


Fig. 3—Stress-Rupture Characteristics at 1200 Degrees Fahr. of 18 Chromium-8 Nickel Tubes After Prolonged Service.

Table V
Rupture Test Characteristics at 1200 Degrees Fahr. (650 Degrees Cent.) of Three 18 Cr-8 Ni Tubes After Prolonged Service

Tube Designation	Service Life Hours	Location of Specimen	Stress Lb./Sq.In.	Rupture Time Hours	Elongation in 1 In. Per Cent	Reduction of Area Per Cent
A	35,000	Fire Side	20,000	145.5	21.0	23.8
		Opposite Fire Side	20,000	316.0	27.0	28.2
		90° to Fire Side	20,000	272.0	22.0	24.0
		90° to Fire Side	22,500	161.0	25.0	28.8
D	97,500	Fire Side	17,000	70.0	8.0	14.6
		Opposite Fire Side	17,000	123.0	16.0	18.9
		90° to Fire Side	17,000	99.5	17.0	16.1
		90° to Fire Side	20,000	49.5	21.0	20.3
E	97,520	Fire Side	17,000	227.0	13.0	13.9
		Opposite Fire Side	17,000	391.0	12.0	15.4
		90° to Fire Side	17,000	339.0	13.0	16.8
		90° to Fire Side	20,000	99.0	19.0	20.4
Bar Stock	W.Q. 2000° F.	—	17,000	308.0	18.0	17.4

Comparative 100-Hour Rupture Strengths:

Tube A	25,000 lb. per sq. in.
Tube D	17,000 lb. per sq. in.
Tube E	20,000 lb. per sq. in.
Bar Stock	20,000 lb. per sq. in.

While these results show appreciable differences to exist in the rupture characteristics from tube to tube and also around the circumference of the individual tubes there was no direct correlation between the rupture or ductility characteristics at 1200 degrees Fahr. (650 degrees Cent.) and the behavior in the flattening test at room temperature. In fact, Tube A, which was brittle in the room temperature flattening test, possessed the highest strength and ductility at 1200 degrees Fahr. (650 degrees Cent.).

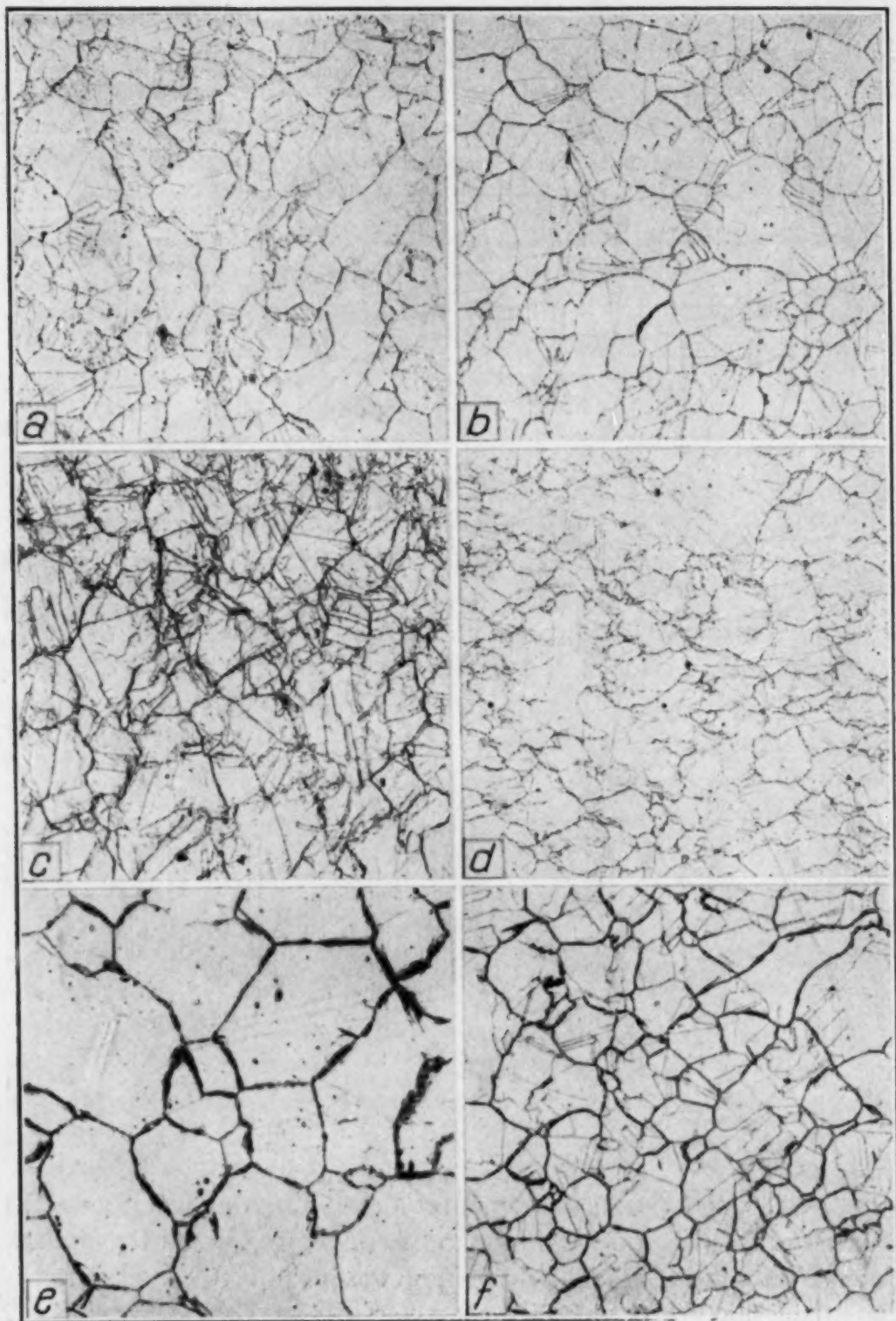


Fig. 4—Microstructures in As-Service Condition. $\times 100$. (a) Fire Side of Tube A. (b) Fire Side of Tube B. (c) Tube C-2. (d) Tube C-1. (e) Tube D. (f) Tube E.

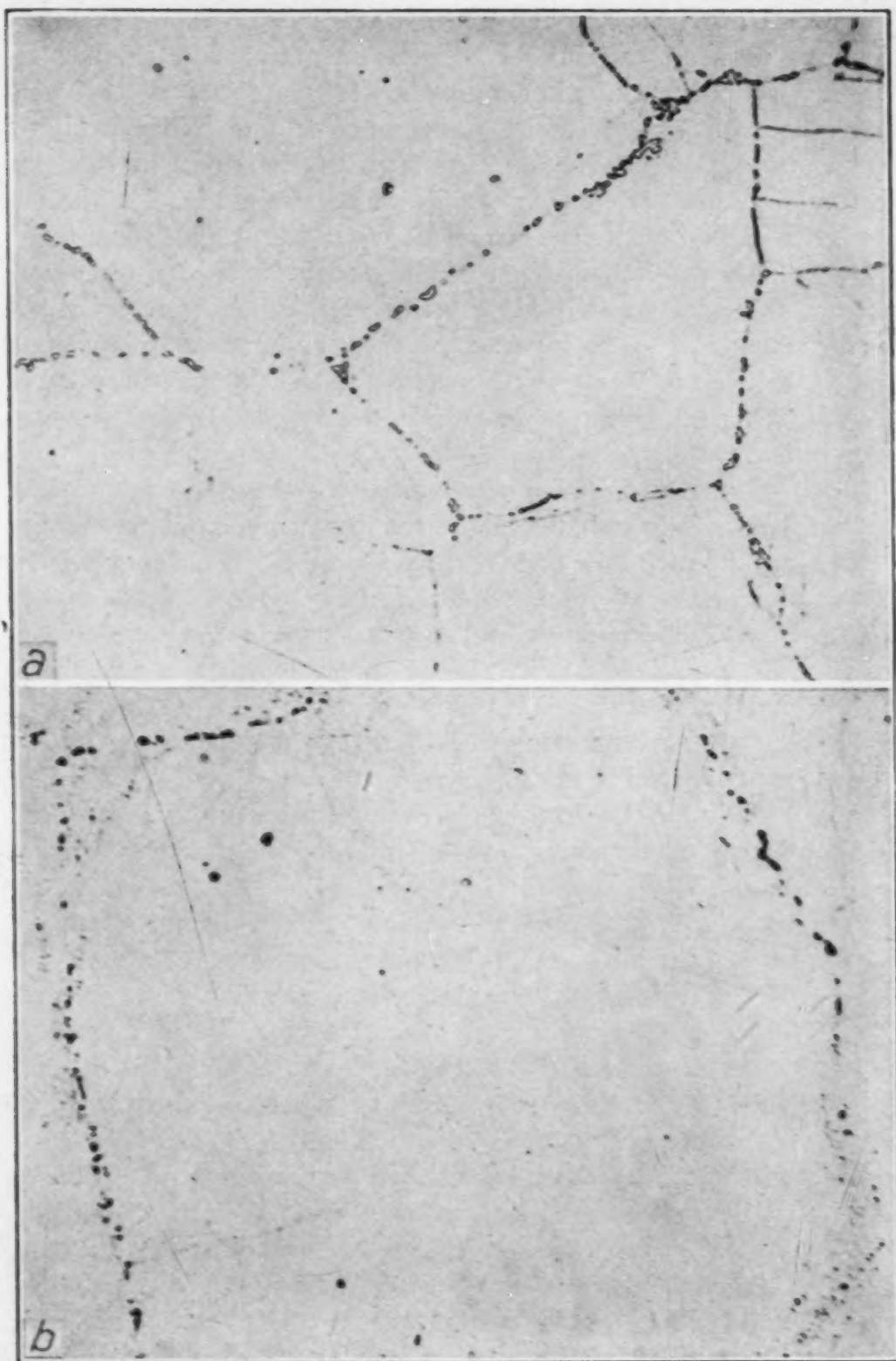


Fig. 5—Characteristics of Grain Boundary Precipitation with Light Electrolytic NaCN Etch. $\times 1000$. (a) Tube B. (b) Tube D.

Microscopic Examination—A thorough metallographic examination was made of each of the sections submitted both in the “as-service” condition and after rejuvenation treatments at 1500 and 1900 degrees Fahr. (815 and 1040 degrees Cent.) as well as at higher temperatures. Representative structures of the tubes in the “as-service” condition are shown at 100 diameters in Fig. 4 while examples of the different type structures observed are given in Figs. 5 to 12 inclusive. A comparison of the various microstructures permits the following generalizations with respect to the degree of structural changes and to the influence of rejuvenation heat treatments:

1. In the “as-service” condition all of the tubes were similar in certain respects, but not necessarily to the same degree, in that

- a. Precipitation was present in the grain boundaries. With a light etch, the grain boundary precipitation was similar around each tube and from tube to tube. The characteristics of this precipitation are shown in Fig. 5.

- b. Twinning was present as shown in Fig. 4.

- c. The grain size was of the same order of magnitude and on the coarse side, being 4 or larger (Fig. 4).

- d. Cold working had occurred on the inner wall, examples of which are given in Fig. 6.

- e. Cracks already present in the tubes or those developed during the flattening tests all followed the grain boundaries, Fig. 7.

2. In the “as-service” condition the following differences existed in the tube sections submitted:

- a. Insofar as outside surface cracking is concerned, Tube C-1 showed more surface cracking, Tubes A, D and E very little, Tube B somewhat more at the fire side and Tube C-2 relatively severe cracking all around the tube (Fig. 8).

- b. All of the tubes showed carburization on the inner wall except Tube C-1 (Fig. 6).

- c. Veining was present to the greatest extent in Tube C-2 and to a lesser extent in Tube A at the fire side (Fig. 4).

- d. Deep etching revealed large precipitated particles in the grain boundaries at the fire side of Tube B and entirely around the circumference of Tube C-2. The same was true to a lesser extent, except at the extreme outer surface on

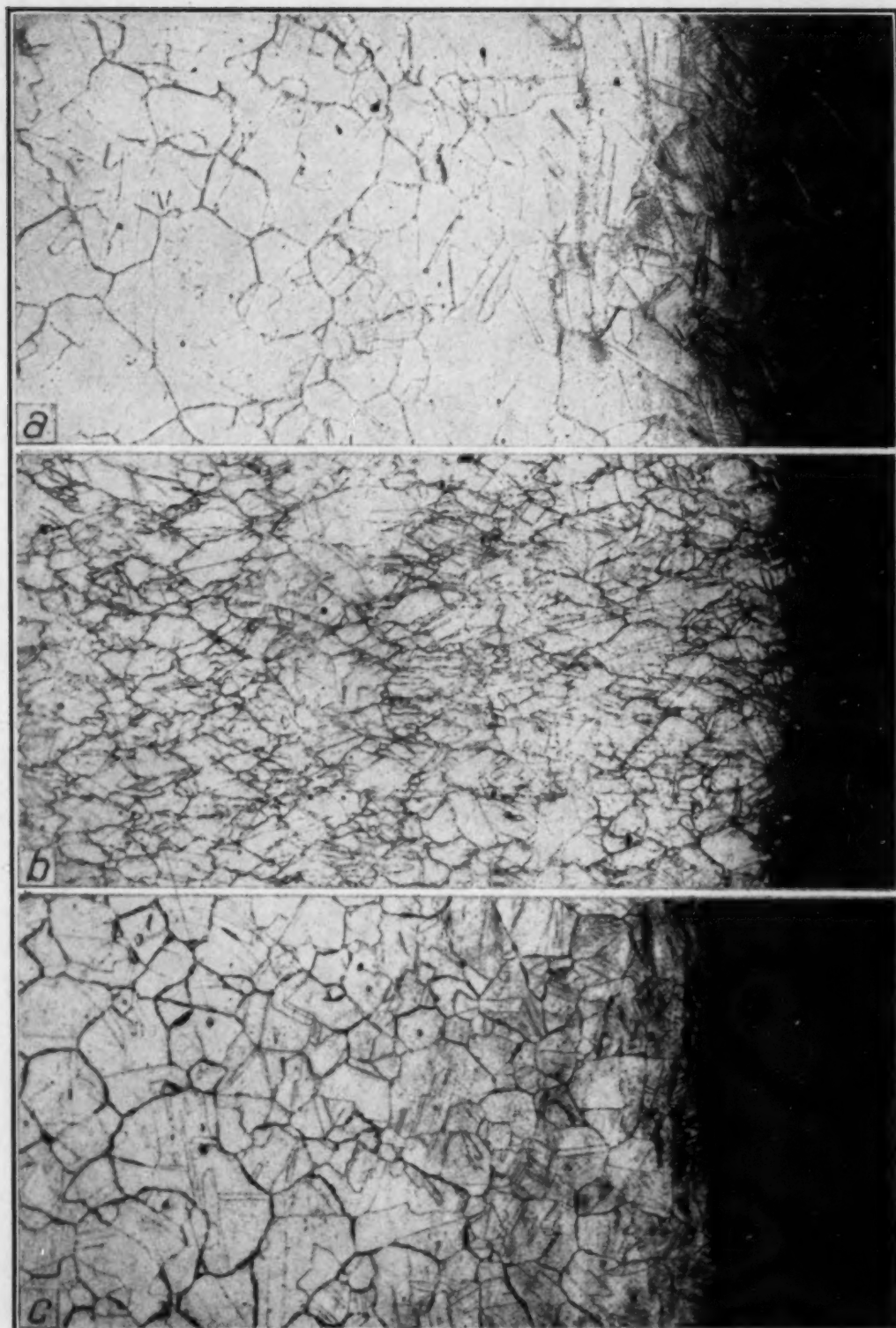


Fig. 6—Examples of Inner Wall Structures. $\times 100$. (a) Tube B. (b) Tube C-1. (c) Tube E.

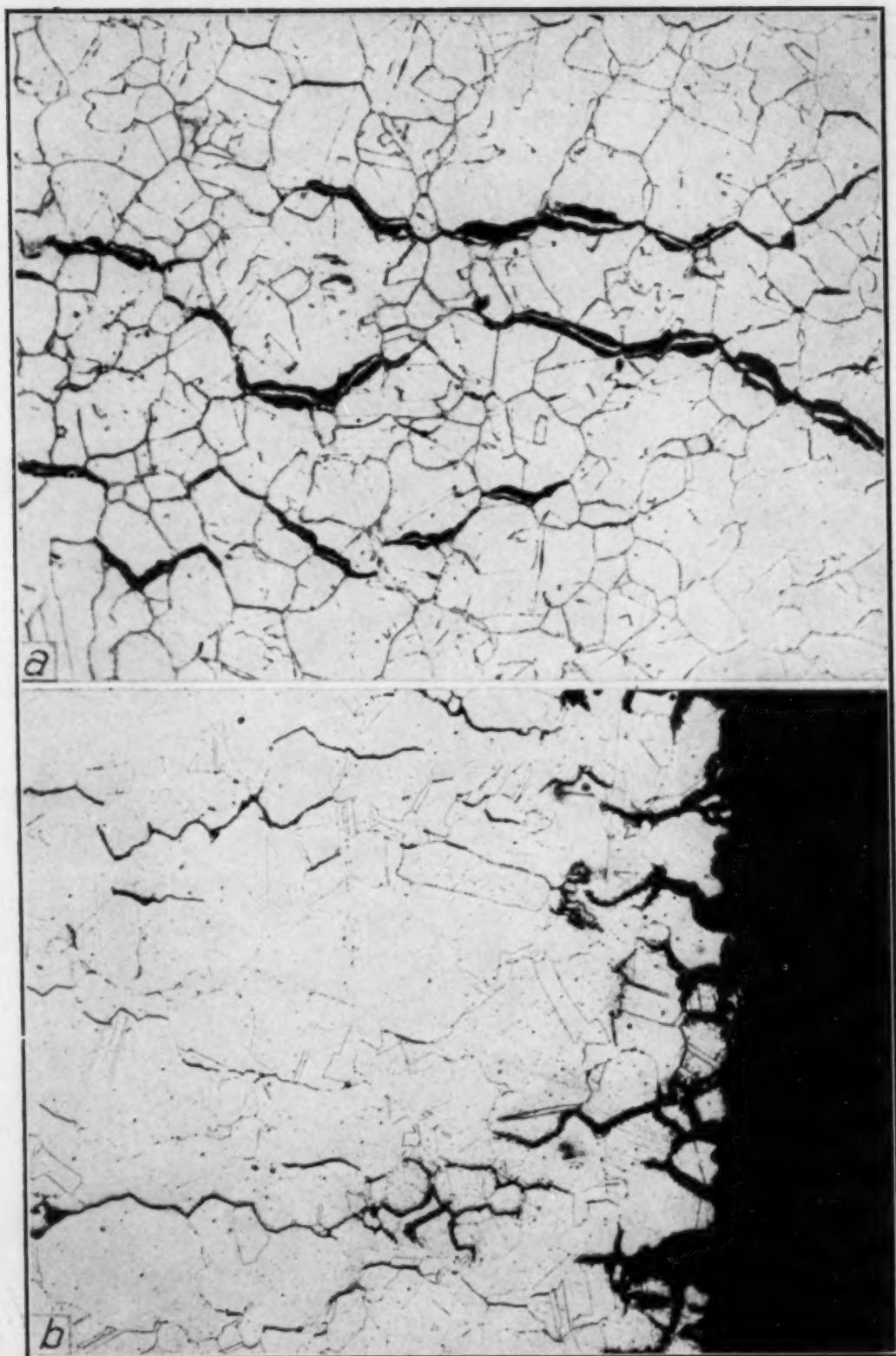


Fig. 7—Intergranular Characteristics of Cracking in 18-8 Tubes. $\times 100$. (a) Tube B—Cracking Developed During Service. (b) Tube C-2—Cracking Developed During Flattening. Specimen Heated at 1900 Degrees Fahr. Prior to Flattening.

the fire side, of Tubes A, D and E, and to even a smaller extent on Tube C-1 (Fig. 9).

3. Rejuvenation treatments at 1500 degrees Fahr. (815 degrees Cent.) produced no visual changes in the microstructures of any of the specimens (Fig. 10).

4. Rejuvenation treatments at 1900 degrees Fahr. (1040 degrees Cent.) exerted variable effects on the microstructures of the different specimens.

a. In all the tubes except Tube C-2 the precipitated constituents were nearly completely eliminated by this treatment (Fig. 11).

b. A "new structure" became visible completely around the circumference of Tube C-2 and to a limited extent on the fire side of Tube B. In Tube C-2 this structure was largely confined to the outer half of the tube wall and was orientated radial to the outside surface and lengthwise to the tube (Fig. 12).

c. This "new structure" could not be removed by solution treatments at temperatures as high as 2400 degrees Fahr. (1315 degrees Cent.).

5. Surface cracking produced during service as well as the cracks produced during the flattening tests followed this "new structure".

From the metallographic examination it would appear that the development of actual cracks and embrittlement of the metal, as determined by the flattening test, are due to alterations of the structure. Tube C-1 was ductile in the "as-service" condition and the metallographic structure, even after prolonged etching, did not show the presence of many large grain boundary particles. In the "as-service" condition Tubes A, B and C-2 did show numerous large grain boundary particles and while these were confined to the fire side of Tubes A and B they existed throughout Tube C-2. All of these tubes cracked during flattening.

After rejuvenation at 1900 degrees Fahr. (1040 degrees Cent.), the structures of Tubes A, D and E became normal and the tubes could be flattened. On the other hand, a so-called "new structure" became visible in Tube B on the fire side and completely around the outer part of the wall of Tube C-2. Neither Tube B or C-2 could be flattened without cracking and the cracks followed this "new structure". The evidence, therefore, points to different degrees of

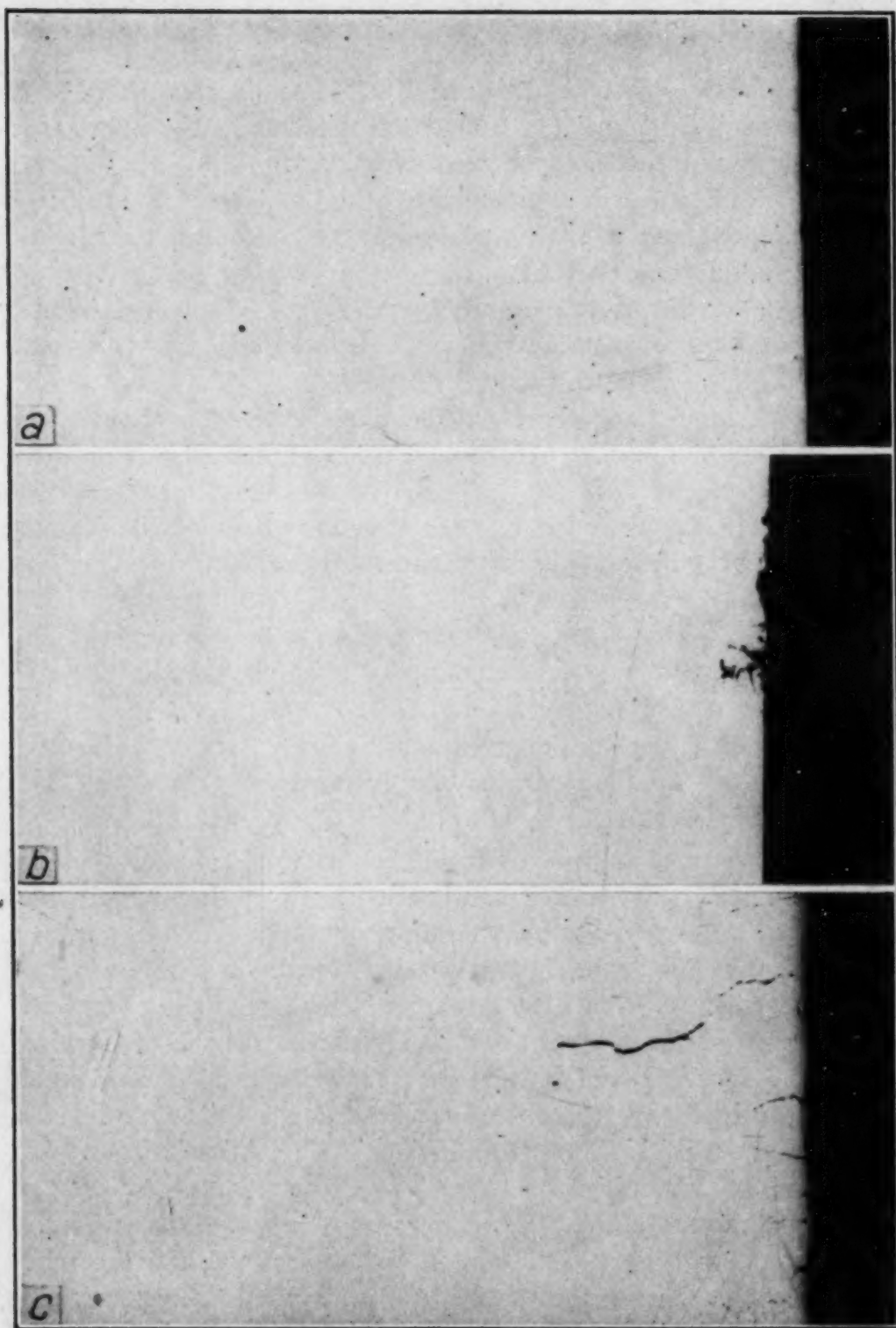


Fig. 8—Varying Degrees of Outside Surface Cracking. $\times 100$. (a) Tube C-1. (b) Tube A. (c) Tube C-2.

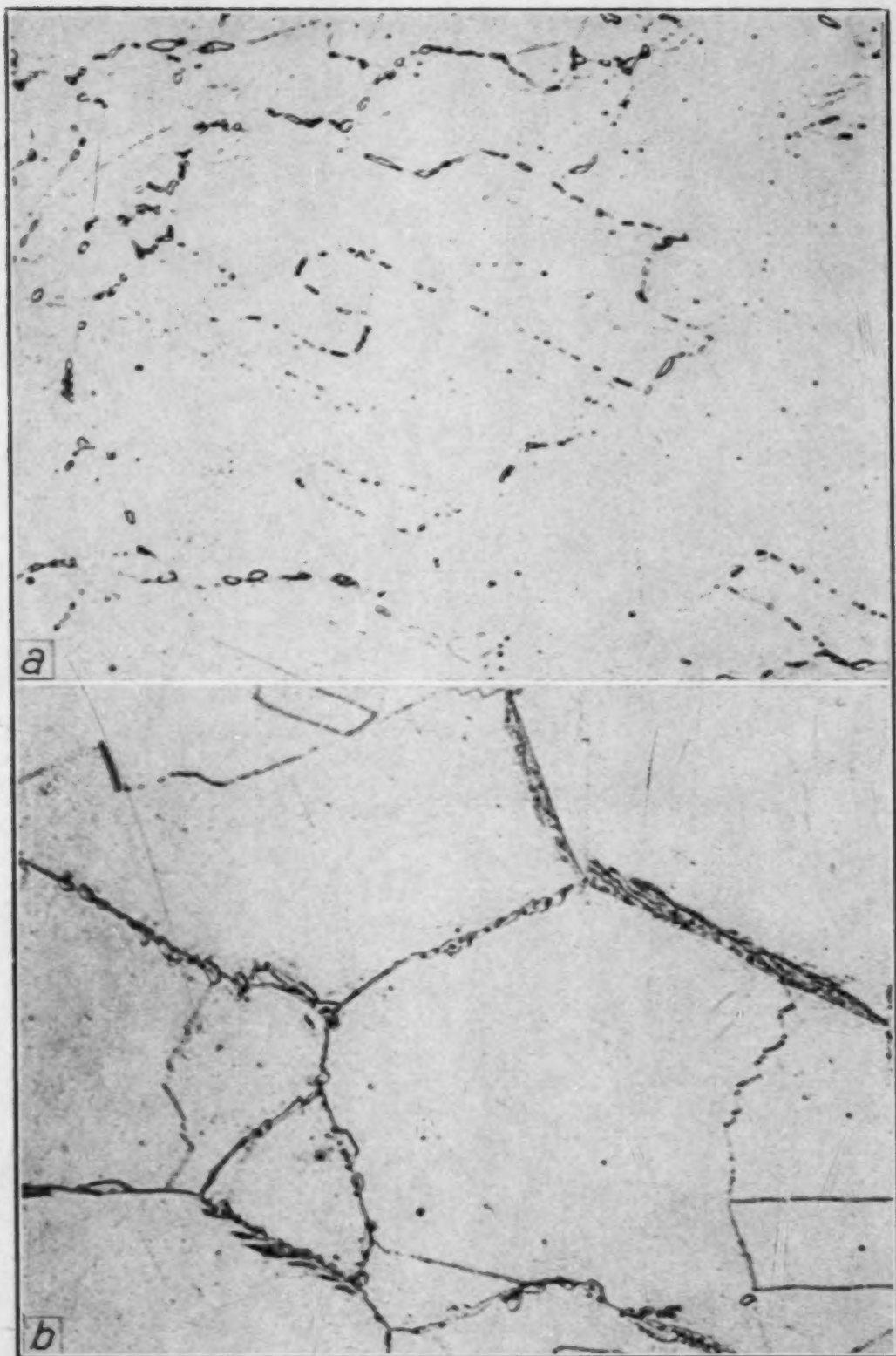


Fig. 9—Characteristics of Grain Boundary Precipitation with Heavy Electrolytic NaCN Etch. $\times 1000$. (a) Tube C-1—Ductile. (b) Tube E—Fairly Ductile. Capable of Complete Rejuvenation.

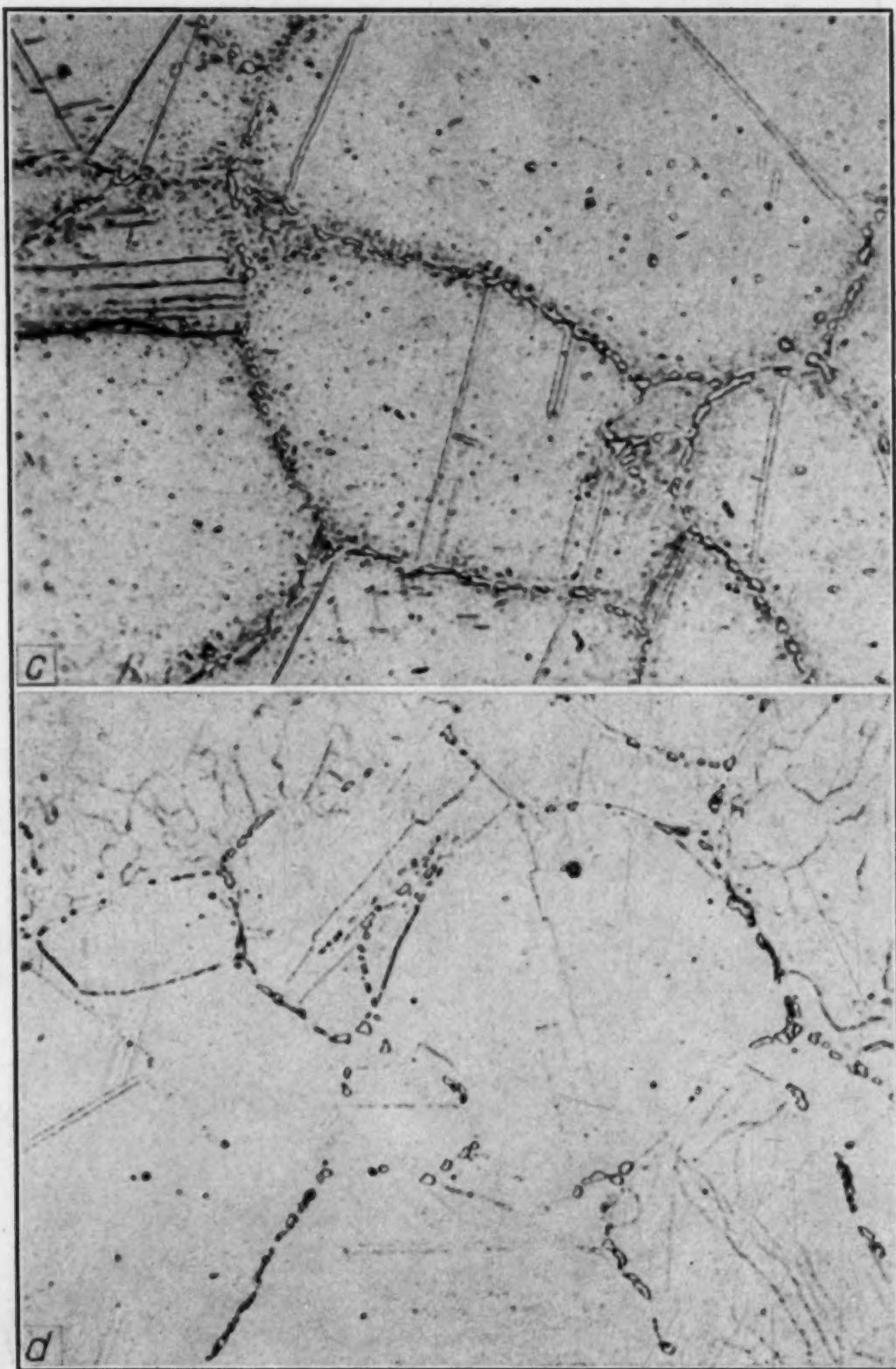


Fig. 9 (Cont.)—Characteristics of Grain Boundary Precipitation with Heavy Electrolytic NaCN Etch. $\times 1000$. (c) Tube B—Erratic Response to Rejuvenation. (d) Tube C-2—Permanently Brittle.

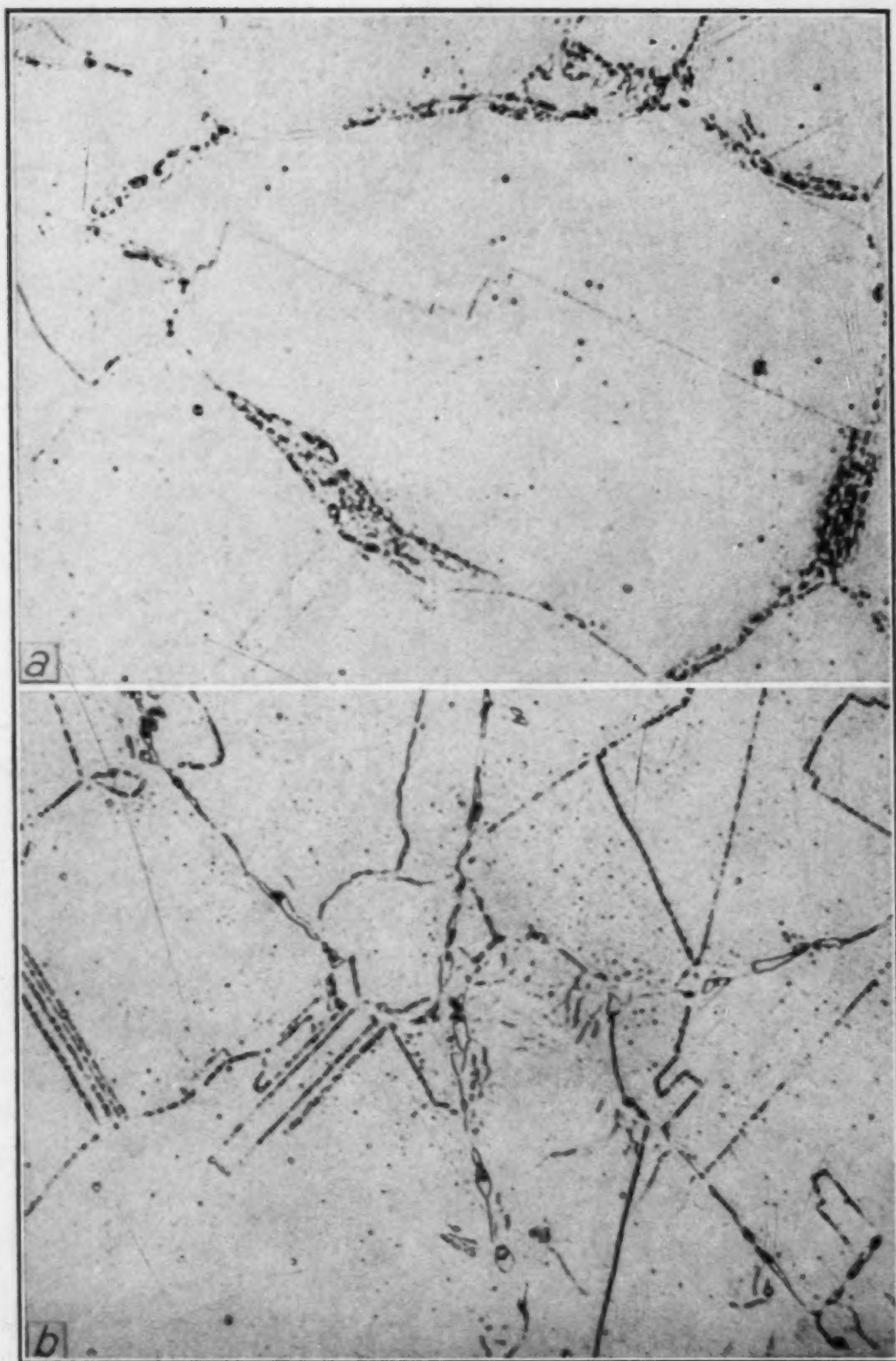


Fig. 10—Influence of Rejuvenation Treatment at 1500 Degrees Fahr. on the Microstructure. $\times 1000$. (a) Tube E. (b) Tube C-2.

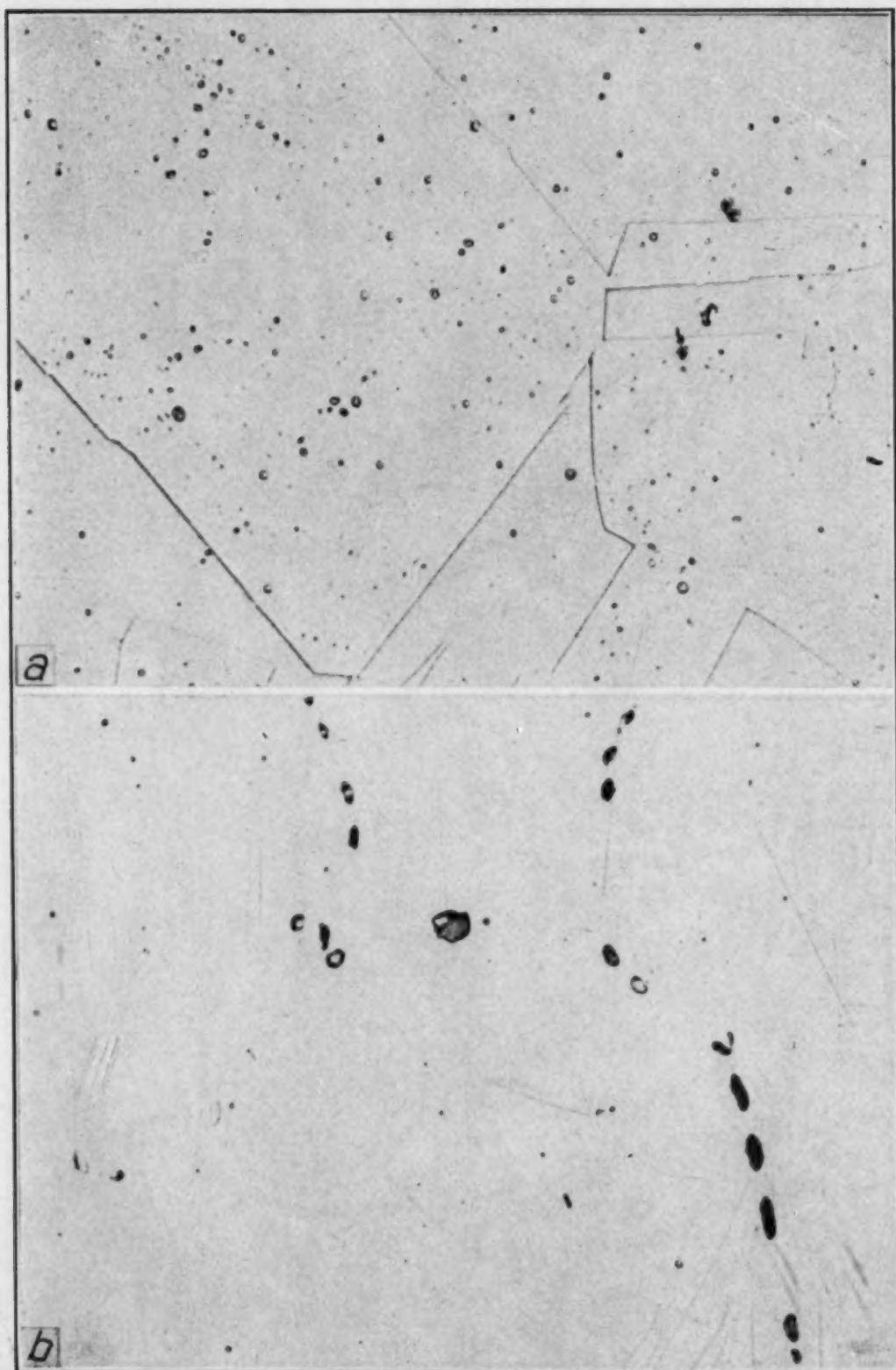


Fig. 11—Influence of Rejuvenation Treatment at 1900 Degrees Fahr. on the Microstructures. $\times 1000$. (a) Tube E. (b) Tube C-2.

deterioration. The grain boundary changes in Tube C-1 had not progressed to a sufficient extent to cause brittleness, while in Tubes A, D and E it had. With these latter tubes, however, this structural change could be removed by heat treatment.

In the case of Tubes B and C-2 it is believed that the embrittlement caused by the grain boundary particles has been further complicated by the development of the "new structure". This condition resulted in poor response to the rejuvenation treatments since the "new structure" apparently cannot be eliminated by heat treatment.

DISCUSSION OF RESULTS

On the basis of these findings it is apparent that the flattening test and microscopic examination are the best means available for determining if 18 Cr-8 Ni steel has undergone changes in its physical characteristics after prolonged service at normal temperatures of 1200 to 1250 degrees Fahr. (650 to 675 degrees Cent.). Chemical composition, within the normal limits, yields no information in this respect for it so happens that the two tube sections showing the maximum variations, Tubes C-1 and C-2, were from different sections of the same tube.

Likewise the visual examination, the hardness survey, the Strauss corrosion test and the room temperature tensile properties did not differentiate between these tubes in the same manner as the flattening tests or microscopic examination. Under certain conditions a fairly definite correlation existed between the magnetic characteristics, the macrostructure, and the flattening properties, but this did not hold true under all conditions, and especially after the rejuvenation treatments. The explanation for these structural changes is not nearly as clear as the correlation between structure and flattening characteristics. Metallographic examination cannot positively identify the large particles revealed in the grain boundaries of the brittle areas by heavy etching. Association of the etching characteristics suggests very strongly that these particles are ferrite. This is also confirmed by the magnetic characteristics. The so-called "new structure" is entirely different from anything previously observed in similar alloys. Actual cracks, when present, terminate in this structure and when cracks are produced, as in flattening, they pass through it.

In the severely cracked areas actual cracks are visible in the

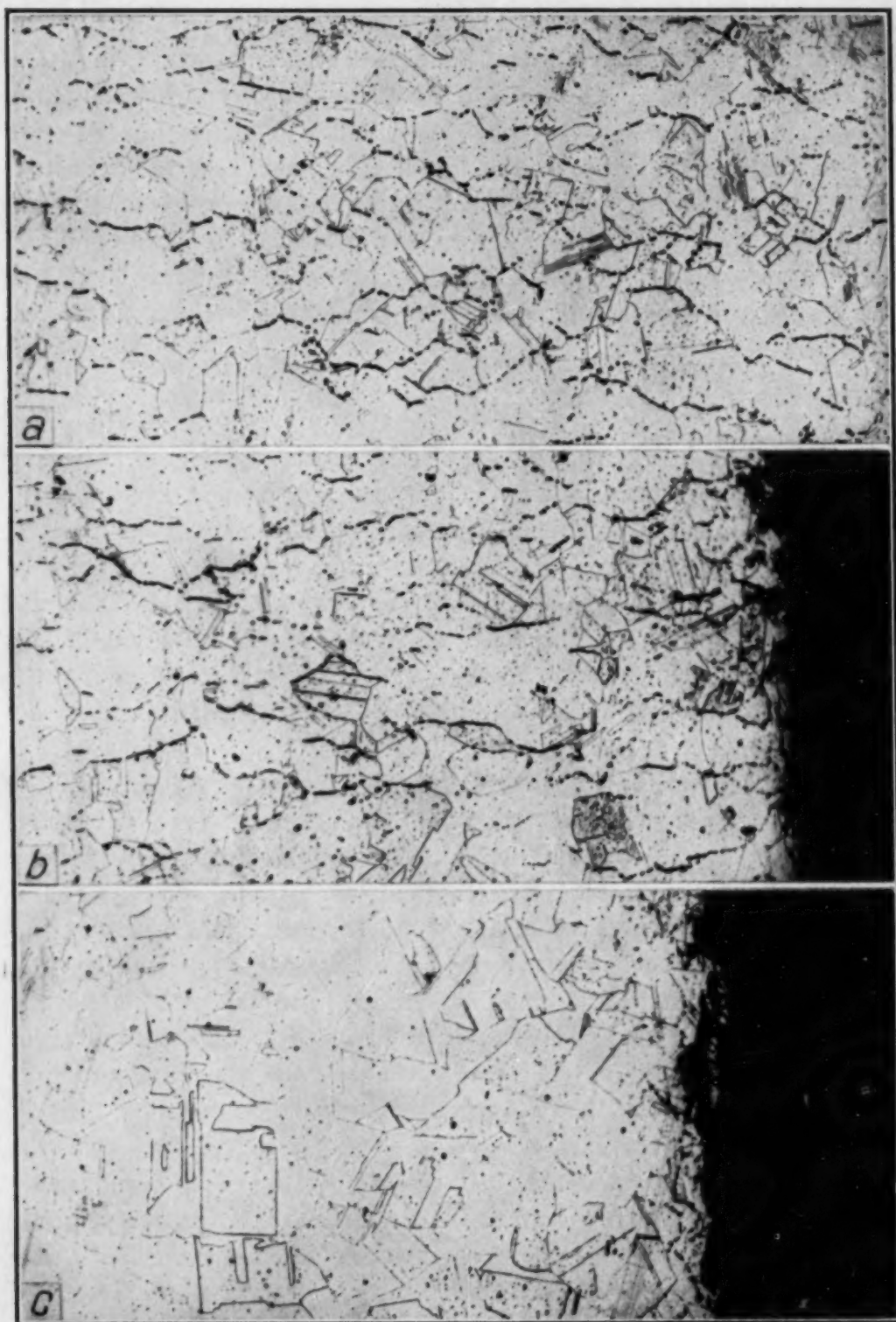


Fig. 12—Microstructures of "New Structure" in Tube C-2 After 1900-Degree Fahr. Rejuvenation. $\times 100$. (a) General Structure. (b) Outside Surface. (c) Inside Surface.

unetched condition which closely resemble it. Since it cannot be eliminated by solution heat treatments it is believed to be very small cracks which surround the large particles present in the grain boundaries of the unheat treated material. These are undoubtedly very small and are invisible under the microscope due to the flowed metal during polishing and are only revealed after etching has removed this flowed metal and has enlarged the cracks.

The orientation of the "new structure" radial to the outside surface and lengthwise to the tube and its restriction to the outer half of the tube wall suggests the following possible explanation of actual failure:

1. Gradual precipitation and growth of particles at the grain boundaries under certain conditions of service. These particles are believed to consist of highly alloyed ferrite which at certain temperatures at least is brittle.

2. Carbide precipitation may result largely because of the decreased solubility of carbon in this highly alloyed ferrite.

3. During heating and cooling of the tube, and other temperature fluctuations, cracks may develop around and in the ferrite areas due to stresses caused by volume changes accompanying their formation and to differences in expansion between the ferrite and austenite. The cracking may be assisted by carbide and other possible precipitants in the grain boundary areas, which tend to set up brittle conditions.

4. The presence of stress raisers at the outer surface, such as "fish scale" and intergranular oxidation, start cracks which propagate themselves through the "new structure" under the combined influence of thermal stresses and service load stresses.

The evidence points very strongly to localized service conditions influencing the alteration of tube metal structure. In the case of Tubes A and B it was localized on the fire side. Likewise Tube C-2 was affected while Tube C-1 was not and yet they were different sections of the same tube.

Insofar as these tubes are concerned the microstructure clearly shows the affected zones. The tube dimensions also indicate that creep was greater on the fire side and the rupture tests indicate the metal to be weaker in this section. The fact that brittleness was restricted to the fire side in two cases suggests higher temperatures at those points, which is equivalent also to a more overstressed con-

dition, both on the basis of mechanical and thermal stresses. Both of these conditions contributed to increased rates of structural alterations. In the case of Tube C-2, the deterioration completely around the tube suggests that the complete circumference of this tube was overheated or else that the tube may have been rotated at various times during its service life. Such information as is available on these tubes indicates that neither corrosion nor oxidation played an important role in the deterioration of these tubes. It is entirely possible, however, that either of these two factors may have hastened the actual failures which occurred in certain areas of certain of these tubes.

CONCLUSIONS

The results obtained permit the following tentative conclusions with respect to the mechanism of failure of 18-8 cracking still tubes in service.

1. Structural alterations occur which are progressive in nature and when they have developed to a certain degree brittle grain boundary conditions result.

2. The degree to which these structural alterations progress and the rate at which they take place are probably largely determined by the actual temperature, stress and time conditions of service.

3. On the basis of the data obtained it is believed the following mechanism is involved:

- a. Particles, believed to be highly alloyed ferrite, grow in the grain boundaries under the combined influence of temperature, time and stress.

- b. Carbide precipitation follows due to the decreased solubility of carbon.

- c. Under certain operating conditions failure can take place microscopically around these ferrite particles due to the magnitude of the stresses present.

- d. When the above conditions have been established, then brittle service failures can occur. Actual operating conditions such as temperature and stress fluctuations and stress raisers on the surface determine the degree to which both particle growth and the cracks must progress before service failures occur.

4. So long as the "new structure", believed to be micro-

scopic cracks, does not form around the precipitated grain boundary particles, ductility can be restored to the tubes by rejuvenation treatments at 1500 and 1900 degrees Fahr. (815 and 1040 degrees Cent.).

5. Service conditions may vary around the circumference and along the length of cracking still tubes. This condition may result in the severe structural alterations being confined to a small narrow portion of the tube; or to conditions where an entire tube can be embrittled.

6. The variations in structural alterations in different portions of a tube are reflected in metallurgical test results. Some of these properties are influenced more than others. For instance, hardness tests show little difference, tensile tests slight differences, while flattening and rupture tests show wide differences between areas with slight and pronounced grain boundary particle growth.

7. The beneficial effect of the 1500-degree Fahr. (815-degree Cent.) rejuvenation heat treatment is believed due to the elimination of stress and to a possible transformation of the precipitated ferrite. It has little influence on the precipitated carbides, as evidenced by the microscopic examination, and yet it imparted immunity to the Strauss corrosion test.

8. Insofar as these particular tubes are concerned, neither corrosion nor oxidation played an important role in their deterioration or failure. It is believed, however, that when the structural changes have occurred, the corrosion resistance is decreased, and on this basis corrosion or scaling may have contributed to the cracking to failure which occurred at certain locations.

While all of the evidence obtained to date substantiates the theory advanced as to the mechanism of failure, additional work is in progress on this general subject and attempts are being made to reproduce those various structural alterations in the laboratory.

ACKNOWLEDGMENT

The experimental work on which this paper was based was sponsored by The Timken Roller Bearing Company, Steel and Tube Division, at the University of Michigan. The investigation was conducted in the Department of Engineering Research of the Univer-

sity, with the assistance of Messrs. E. E. Reynolds and M. Goldenberg.

The authors are indebted to the oil companies whose co-operation made the investigation possible and to The Timken Roller Bearing Company for permission to publish the results.

DISCUSSION

Written Discussion: By C. P. Miller, head of Materials Laboratory, Standard Oil Company of California, Richmond, Cal.

The writer has directed the examination of a considerable number of 18 Cr-8 Ni cracking still tubes which had failed in the manner described by Messrs. Clark and Freeman. The first failure occurred in 1932 after about 22,000 operating hours. In this connection, it is of interest that, although failures have continued since then at a progressive rate, a few tubes of the same vintage are still in service after a life of nearly 100,000 hours.

In all, we have made detailed examination of about 25 failed tubes. Our findings are in general agreement with those of the authors. Embrittlement as exhibited in the flattening test was invariably associated with the failures. In some cases, this embrittlement could be removed by the so-called "rejuvenation" heat treatment; in other words, rejuvenation was ineffective. There was also a noticeable difference in the microstructure of brittle and ductile tubes and in magnetic permeability, which was substantially higher in brittle tubes.

There was no significant difference in chemical composition, surface appearance, hardness, impact strength, carbon pick-up and resistance to the Strauss solution. There was also no difference in the tensile properties of longitudinal specimens; the strength of both ductile and brittle tubes was slightly greater than that of new material and the elongation was somewhat less. On the other hand, the circumferential strength of brittle tubes, as determined by bursting tests, was less than half that of ductile tubes and there was no measurable elongation. This points to an extreme orientation of embrittlement which has been confirmed by micro-examination of the structure.

From the first, embrittlement was recognized as the primary cause of the failures. It was also realized that an accompanying stress was necessary to produce actual cracking. This stress was and still is attributed to the effect of the temperature differential through the tube wall during firing. The outside of the tube tends to expand more than the inside and a certain amount of plastic flow or "upsetting" occurs. At shutdown, the differential no longer exists and there is a reversal of stress which puts the outer fibers of the tube in tension. A ductile tube is amply able to withstand this stress while a brittle tube will fail by cracking.

It is reasonable to believe that internal fluid pressure during operation was of negligible influence in causing cracks to form. Otherwise, a crack once started would continue through the wall to complete rupture while the tube was on stream. This has not been the case as, except for a few tubes in which

other factors were involved, all cracks have been found by inspection at shut-down. Furthermore, in nearly every case, they have only extended to the neutral axis which is where they should stop if they were formed by stress reversal as described above.

Embrittlement was first attributed to intergranular corrosion and then to stress-corrosion of the type described by A. E. White, C. L. Clark and R. L. Wilson in their paper, "The Rupture Strength of Steels at Elevated Temperatures," appearing in the March 1938 *TRANSACTIONS* of the American Society for Metals. It was evident, however, that both of these explanations were in conflict with the phenomenon of rejuvenation. We finally adopted a theory of structural change analogous to that offered by the authors of the present paper. In it, we postulated that this change produced embrittlement and was reversible upon proper heat treatment, thus permitting "rejuvenation". Under more prolonged exposure or more adverse conditions, the tube suffered actual structural damage such as the formation of submicroscopic cracks and, therefore, could not be "rejuvenated".

There are unquestionably at least three factors involved in this structural change—temperature, stress and time. Unfortunately, even now little or nothing of a quantitative nature is known regarding their action in causing failures. Temperature is obviously critical as embrittlement and cracking are almost always confined to the fire side of the hotter tubes. There must, however, be a temperature below which no damage occurs in order to account for the existence of ductile tubes after service of 100,000 hours or more. This temperature is still unknown. The relation between temperature and time required for embrittlement is also still undetermined. Is there a temperature at which embrittlement occurs in a few hours or is it always a slow process, measured in terms of months or years? If a tube has been somewhat embrittled by exposure to, say, 1400 degrees Fahr. (760 degrees Cent.), is it thereafter more susceptible to damage at a lower temperature? Is there an upper temperature limit beyond which no harm is done provided the tube has sufficient strength to withstand the fluid pressure?

When we introduce the third variable of stress, the confusion is even greater. Just how critical is the influence of stress? Is it so critical that the difference between longitudinal and hoop stress due to fluid pressure is sufficient to cause the orientation of embrittlement previously mentioned? Is a high stress at a relatively low temperature as damaging as a low stress at a higher temperature?

Also, assuming that the properties of straight 18 Cr-8 Ni are such that tubes of this composition are always liable to damage under conditions imposed by economical operation, is there any way, short of prolonged large-scale trials, to determine whether tubes of modified composition would be better? In other words, is there any reliable accelerated test from which the behavior of tubes could be predicted?

The authors' explanation of the mechanism of failure is quite convincing and is in agreement with the known facts. The tubes could fail in the manner described and they probably do. There is nothing in the paper, however, to indicate that embrittlement has been induced by the authors under controlled condi-

tions. It would be very worthwhile if this were done as it would not only confirm the correctness of their theory, but would furnish much of the information needed to answer the questions listed above. These answers, it is needless to say, are of practical interest to the user of 18 Cr-8 Ni cracking tubes as they would enable him to control operation so as to obtain maximum tube life, and also be sure that he was buying tubes of the most suitable material.

Written Discussion: By E. R. Wilkinson, Standard Oil Company of Louisiana, Safety Inspection Dept., Baton Rouge, La.

This paper on the failure of 18 Cr-8 Ni cracking still tubes brings out some very unique conclusions regarding structural alterations which occur within the metal, and which may be corrected by proper heat treatment, if the condition is detected before it has progressed beyond a certain phase of transformation. It has been known for quite some time that some 18 Cr-8 Ni material, which had been in service for long periods of time, could be revived or rejuvenated by heat treatment. On the other hand, we have seen material of the same composition and which apparently received similar treatment in service which could not be restored to a sound and ductile metal by heat treating. Because of this erratic response to heat treatment, we could not always be sure that the material was capable of rejuvenation, nor could we be sure of the extent to which the heat treatment would be beneficial. The investigations covered in this paper have given us some of the answers to these questions; however, there are still several questions which arise concerning the practical applications of the results and conclusions which are presented.

It is stated in the paper that the structural changes within the metal are due to a certain combination of time, temperature, and stress; and that once these changes reach the point where submicroscopic intergranular cracking occurs, the metal can no longer be restored to its original ductile state. When the additional laboratory tests which are now in progress are completed, will there be enough data from the investigations so that a correlation can be made of the time, temperature, and stress relationships which could be used as a guide for removing a tube from service for heat treatment? Such an analysis might also help to determine whether or not a definite service hour limitation should be maintained and, if so, what this limitation should be under certain specific conditions of temperature and stress; or is it felt that no such correlation would be of any particular value when considering that one of the tubes which was tested was apparently in good condition on one end and in a very poor state on the other end? No doubt the two ends of this particular tube were subjected to wide temperature variations in service. Such conditions would be extremely difficult to discover without the aid of numerous metal thermocouples and very elaborate instrumentation.

Quite often we desire to make test runs on furnaces and operate them at higher temperatures, or higher temperatures and pressures, for a short period of time in order to determine operational limitations. Before proceeding, however, one of the first and foremost questions always is whether any harm will be done to the tubes by overheating them for a short period of time at temperatures considerably in excess of those which have long been established as satisfactory and safe. It is indicated in the paper that this change which takes

place in tube metal structure can likely be contributed either to localized overheating or to a general overheating of the metal over a period of time. Here again this time factor is quite important. We are wondering if the data which have been and are now being obtained will enable us to formulate any limitations in time and temperature for periods of overheating, such as those mentioned, so that the metal would not be detrimentally affected?

It is noted that there was no mention of the operating pressures to which the tested tubes had been subjected. Inasmuch as two of the tubes which had been in service over ninety thousand hours were still fairly ductile, it would be very interesting to know something of the pressure conditions. If possible, it would also be desirable to know something about the general type of tube and header installations, and what sort of maintenance and inspection treatment these tubes received during their ninety thousand hours of service. This information may be of particular interest to anyone who has established service hour limitations on furnace tubes which are subjected to high pressures.

The question of furnace tube design has long been a very debatable one. Some methods employ the use of creep stress data, some use stress rupture data, while others use short-time tensile stress with a high factor of safety. It would be of interest to know whether or not the authors of this paper feel that the results of their investigations with this 18-8 material will throw any additional light on which of the above methods might be considered the most practical. If this cannot be determined, will their results give us any indication as to whether creep stress or stress rupture data are the most reliable to be used in the design of furnace tubes.

It is quite interesting to know that the flattening and microscopic tests are the best means of determining if 18-8 chromium-nickel steel has undergone structural changes discussed in this paper. Both of these tests are quite practical and they can be very readily made with only a small amount of material.

The investigations conducted on this subject by the authors of this paper are typical of the very fine work which they have done in all of their research work for the development of alloys for high temperature service. We in the petroleum industry should feel highly indebted to them for the work which they have done in this connection. We are quite anxious to know a little more about the behavior of 18-8 chromium-nickel tubes operating at metal temperatures up to the order of 1600 to 1700 degrees Fahr. (870 to 925 degrees Cent.) and we trust that, before too long, they will be able to give us good information in this regard.

Oral Discussion

GLENN R. INGLES:¹ I would like to ask of the authors if the tubes studied showed any carburization after the long service period? Also, what is the effect of time on the formation of the discussed structure? Could this structure account for failures of cracking tubes in service for as short times as 200 to 300 hours?

H. S. AVERY:² Was it necessary to go to microradiography to detect the

¹Metallurgical consultant, Cook Heat Treating Co., Houston, Texas.

²Research metallurgist, American Brake Shoe Co., Mahwah, N. J.

cracks, or was it possible to get evidence of them by dark field illumination under high magnification?

Authors' Reply

We greatly appreciate the discussion which has been offered on this paper and are pleased to know that the experiences of certain of the refineries are in agreement with our results. Our findings to date are confined to the various conditions which may be encountered in this austenitic steel after high temperature service. It is hoped, on the basis of laboratory work now in progress, that definite conclusions can be reached with respect to the time-temperature relationships under which the submicroscopic cracking occurs. If this can be done, then, as Mr. Wilkinson suggests, definite service life conditions could be established for the various temperatures, as well as permissible time periods of overheating.

It is true, as Mr. Wilkinson states, that in service the temperature may vary along the length of the tube and around the circumference at any given point. It is believed, however, that in each cracking still the hotter zones can be established and the service life of the tube should be based on these higher temperatures.

In further reply to Mr. Wilkinson's questions, the operating pressure in the tubes which had been in service for 97,000 hours was 900 pounds per square inch, which on the basis of Barlow's formula gives a fiber stress of 4200 pounds per square inch. It so happens that this stress is in exact agreement with the stress required at 1200 degrees Fahr. (650 degrees Cent.) for a creep rate of 0.01 per cent per 1000 hours. Even after this long service life these tubes were removed, not because of bulging, but because of wall thinning due to ID corrosion. This in turn indicates that the creep values are a too conservative basis of design for this type of service. To us, it would appear that the stress rupture values are a more suitable basis for the design of cracking still tubes.

Mr. Miller has given many interesting results concerning the tubes which he has examined after service and has also raised many important questions which at the present time cannot be definitely answered. It is hoped, however, that the results from laboratory tests now in progress will shed additional light on these questions.

In reply to Mr. Ingles' question most of the tubes considered in this investigation did show a slight amount of carburization on the inner surface. We do not as yet definitely know the effect of time on the submicroscopic cracking but we believe 18-8 tube failures of the order of 200 to 300 hours are probably due to excessive overheating.

In answer to Mr. Avery, it was not necessary to use microradiographic means to detect these cracks but this method was employed for further substantiation.

This work is being continued and the investigation is being extended to cover 18-8 tubes after service at the more elevated temperatures. It is hoped that in due time more complete information can be obtained concerning the mechanism of failure of these, as well as other type, austenitic steels.

THE RATE OF DIFFUSION OF MOLYBDENUM IN AUSTENITE AND IN FERRITE

BY JOHN L. HAM

Abstract

The coefficients of diffusion of molybdenum in pure austenitic and ferritic iron-molybdenum alloys and in pure austenitic iron-molybdenum-carbon alloys are reported. The coefficient of diffusion of molybdenum is found to be much greater in ferrite than in austenite. The coefficient of diffusion of molybdenum in austenite is found to be slightly increased by carbon.

The composite nature of the diffusion coefficients of binary substitutional solid solutions is discussed.

Diffusion and partition data are used as the basis for an explanation of the effect of molybdenum on the shape of the S-curve.

Equations expressing relative rates of homogenization in terms of coefficients of diffusion are presented.

INTRODUCTION

THOSE who are seriously attempting to account for the rates of metallurgical phenomena well recognize the need for accurate data on rates of atomic diffusion in the solid state. Rates of processes such as carburization and homogenization, occurring under simple conditions, can be explained from a knowledge of the diffusion coefficients involved. However, an explanation of more complex phenomena such as creep, phase change, and precipitation, requires a knowledge of other types of fundamental data as well as a knowledge of diffusion coefficients. Rates of diffusion, like electrical conductivity and other phenomena, must be accounted for in any theory of the solid state of matter and therefore are useful in setting up and testing such theories.

In the practical as well as the theoretical application of data concerning the rate of diffusion of molybdenum in iron, it is necessary to know the effect of temperature and atomic configuration. The effect of these variables has been determined in the experiments about

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to be described. These experiments are intended to supplement previous experiments (1)¹ concerned with the rate of diffusion of carbon and the effect of molybdenum thereon. They are part of a program which includes a study of the partition of molybdenum between the ferrite and carbide phases in steel and which is intended to provide an insight into the mechanism by which molybdenum affects the hardenability and other properties of steel.

Hardenability, or the ability of large masses of steel to harden uniformly, depends directly on the rate at which the steel in the austenitic condition must be cooled to prevent its transformation at high temperatures; for transformation at high temperatures leads to the formation of pearlite or bainite, constituents which are soft relative to martensite, which is formed when transformation occurs at low temperatures. Since the rate at which a body of steel can be cooled at some point below the surface is limited by the thermal conductivity of the steel (a property which is not significantly affected by alloying elements), any major improvement in hardenability obtained by the addition of a given element must result from the ability of this element to retard the rate of transformation of austenite at temperatures where the products of transformation are soft. The effect of an alloying element on the rate of transformation at constant temperature is conveniently represented by the Bain S-curve, which shows contour lines for the beginning and end of transformation on a graph with temperature as the ordinate and the logarithm of the time as the abscissa.

It early became apparent (1), (2) that the ability of alloying elements such as molybdenum to retard the rate of decomposition of austenite could not be explained by their effect on the rate of diffusion of carbon in austenite. That the effect depended in some way on the rate of diffusion of the alloying element itself seemed likely, and the magnitude of the rates of diffusion of manganese and nickel (3), (4) seemed to support this assumption. To test the validity of this assumption it is imperative that the partition of the alloying element between ferrite and carbide during and after transformation be known. Such information on the partition of molybdenum has been obtained already (5), (6). It appears from this earlier work that the carbides precipitating during the transformation of austenite to pearlite at a given temperature have initially a much higher molybdenum content than, and a crystal structure differing from,

¹The figures appearing in parentheses pertain to the references appended to this paper.

that of the carbides of the ordinary cementite type which form during tempering of martensite at the same temperature. At the expense of the surrounding ferrite, both types of carbides apparently increase their molybdenum content after formation, although the molybdenum content of the carbides formed from austenite is always the greater. The consequences of these findings and their bearing on the conditions under which the diffusion process proceeds will be discussed after a presentation of the results of the diffusion experiments and after a discussion of some general aspects of the diffusion process.

EXPERIMENTS AND RESULTS

The experimental method for determining coefficients of diffusion was described in a previous paper (1). Briefly, the method consists of placing in intimate contact a pair of specimens containing different amounts of molybdenum, holding them at constant temperature for an appropriate length of time, and chemically analyzing* chips cut at various distances from the interface. The specimens were prepared from electrolytic iron and pure molybdenum by melting in vacuum and, in some cases, casting in vacuum. After considerable forging, the samples were homogenized at 2300 degrees Fahr. (1260 degrees Cent.) for periods ranging from 75 to 100 hours. In nine of the experiments where the diffusion coefficients in austenite were to be determined, the temperature and composition of the specimens were selected so that the specimens remained entirely austenitic during diffusion.

In the two experiments where the coefficients of diffusion in ferrite were to be determined, conditions were such that the specimens remained entirely ferritic during diffusion.

Treatment of the diffusion data for molybdenum was somewhat simpler than that for carbon (1) since the coefficient of diffusion was found to be essentially independent of molybdenum content in the range studied (0 to 6 weight-per cent molybdenum). The boundary value problem is that of the infinite bar with initial concentration c_1 and c_2 atomic per cent, and its solution is

$$\frac{c - c_2}{c_1 - c_2} = \frac{1}{2} \left[1 - \operatorname{erf} \left(\frac{x}{2\sqrt{Dt}} \right) \right]$$

*In Experiment 8 (Fig. 3) the chemical analyses were supplemented by precise X-ray measurements of the lattice parameters of some of the samples. The X-ray method was essentially the same as that used by Bowman (6).

where c is the concentration in atomic per cent molybdenum at any distance x (centimeters) from the interface at a time t (seconds) after the start of diffusion.

D is the diffusion coefficient in centimeters squared per second and $\phi(Z)$ is defined as

$$\phi(Z) = \frac{2}{\sqrt{\pi}} \int_0^Z e^{-w^2} dw$$

where w is an integration variable. Thus when $\frac{c - c_2}{c_1 - c_2}$ is plotted on probability paper (7) as a function of x , a straight line is obtained whose slope is $\frac{1}{2\sqrt{Dt}}$. When D is constant this method is superior to that of deriving D from one or more individual points on a plot of c against x on linear graph paper, since only one D value, determined by all the experimental points, is obtained. If D is not constant a curve is obtained and the Matano method (1) should be used.

In calculating atomic per cent molybdenum from weight-per cent values, the carbon atoms were neglected because, in the derivation of the diffusion equation, the rate of flow is considered to be proportional to the concentration gradient expressed as mass per unit volume. In a substitutional solid solution whose lattice parameter is being considered constant, the mass of molybdenum per unit volume is equivalent to the percentage of lattice points occupied by molybdenum and does not necessarily depend on the number of carbon atoms present, since they are dissolved interstitially.

To simplify presentation and calculation, the experimental data are given in the form of points on a plot on linear graph paper of $\frac{c - c_2}{c_1 - c_2}$ against $\frac{x}{\sqrt{t}}$ in $\text{cm}/\sqrt{\text{sec}}$. See Figs. 1 to 5 inclusive. The curves are the true probability curves represented by the best straight line through the experimental points plotted on probability paper. The D values and other data are given in Table I. See page 336.

In Fig. 6 it can be seen that the coefficient of diffusion of molybdenum is much greater in ferrite than in austenite; at 2300 degrees Fahr. (1260 degrees Cent.) it is about 80 times greater and at 1700 degrees Fahr. (925 degrees Cent.) about 90 times greater. Carbon slightly increases the coefficient of diffusion of molybdenum in austenite.

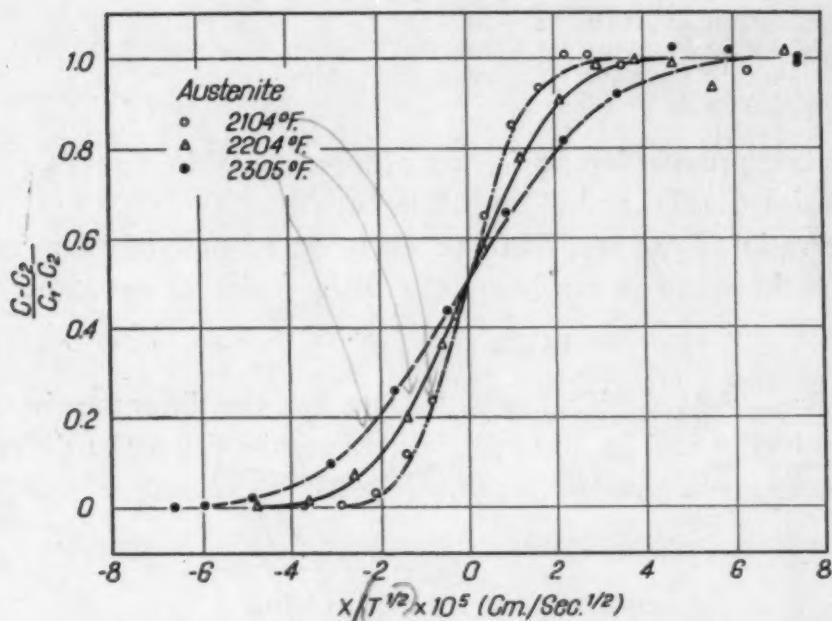


Fig. 1—Diffusion Curves for Molybdenum in Austenite. Experiments 1, 2 and 3.

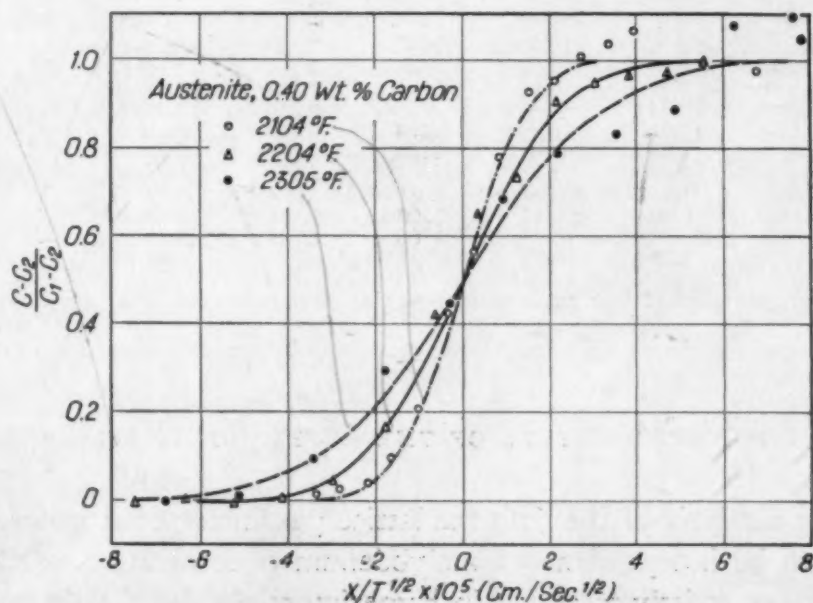


Fig. 2—Diffusion Curves for Molybdenum in Austenite. Experiments 4, 5 and 6.

It has been assumed that the D values can be represented by equations of the type $D = Ae^{-Q/RT}$, where A , Q , and R are constants. That this is approximately the case can be seen by inspection of Fig. 6, in which D is plotted on a logarithmic scale as a function of the reciprocal of the absolute temperature. The equations of the straight lines are:

Molybdenum in Austenite

Molybdenum in Austenite Containing 0.40
Weight-Per Cent Carbon

Molybdenum in Ferrite

$$D_A = 0.068 e^{-59,000/RT}$$

$$D_{AC} = 0.091 e^{-59,000/RT}$$

$$D_F = 3.467 e^{-57,700/RT}$$

where Q is expressed in calories per gram-atom, T in degrees Kelvin, R in calories (1.99), and D and A in cm^2/sec .

The ratio of the coefficient of diffusion of molybdenum in ferrite to that in austenite can be expressed by a similar equation:

$$D_F/D_A = 51.3 e^{1300/RT}$$

Of theoretical significance is the fact that the difference in the D values in ferrite and in austenite, largely results from the difference in the corresponding A values; the Q values do not differ greatly.

Table I
Diffusion Data, Molybdenum in Iron

Experi- ment	Initial Atomic Per Cent Molybdenum— C ₁ C ₂		Weight Per Cent Carbon*	Temperature Degrees Fahr.	Time Sec. x 10 ⁻⁶	Phase	D x 10 ¹⁰ Cm ² /Sec.
1	0.532	< 0.005	< 0.03	2305	0.360	Austenite	3.12
2	0.540	< 0.005	< 0.03	2204	0.791	Austenite	1.42
3	0.550	< 0.005	< 0.03	2104	1.577	Austenite	0.613
4	0.540	0.145	0.38	2305	0.360	Austenite	3.62
5	0.520	0.145	0.44	2204	0.791	Austenite	1.68
6	0.530	0.145	0.47	2104	1.577	Austenite	0.73
7	1.470	0.000	0.25	2301	0.508	Austenite	4.11
8	3.607	1.928	< 0.03	1708	1.732	Ferrite	1.23
9	0.585	0.004	< 0.03	2300	0.346	Austenite	2.46
10	1.475	< 0.005	0.35	1708	1.732	Austenite	0.0189
11	3.629	1.958	< 0.03	2301	0.508	Ferrite	216.0

*These are averages of four analyses representing concentration of each specimen before and after diffusion.

THE COMPOSITE NATURE OF THE DIFFUSION COEFFICIENT

In the analysis of the data the lattice parameter of iron has been assumed to be independent of molybdenum concentration. This is, of course, not strictly true, for in Experiment 8 (Fig. 3) the molybdenum content of some of the samples was evaluated by measuring the lattice parameter. The relationship between the lattice parameter of ferrite and its molybdenum content has recently been accurately determined (6).

By a derivation of the diffusion equation based on the percentage of lattice points on adjacent crystal planes occupied by molybdenum atoms, it can easily be shown that D is the product of two more fundamental quantities, namely, the square of the jump dis-

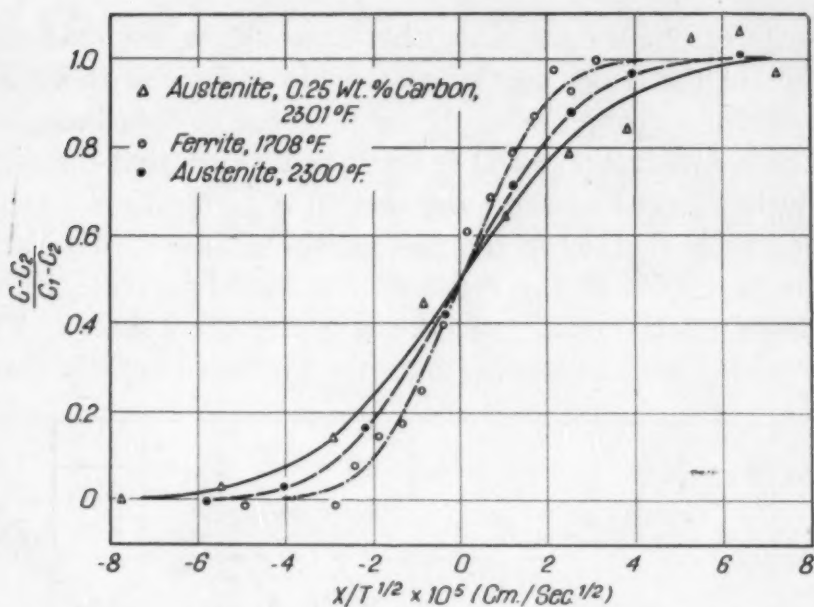


Fig. 3—Diffusion Curves for Molybdenum in Austenite and Ferrite. Experiments 7, 8 and 9.

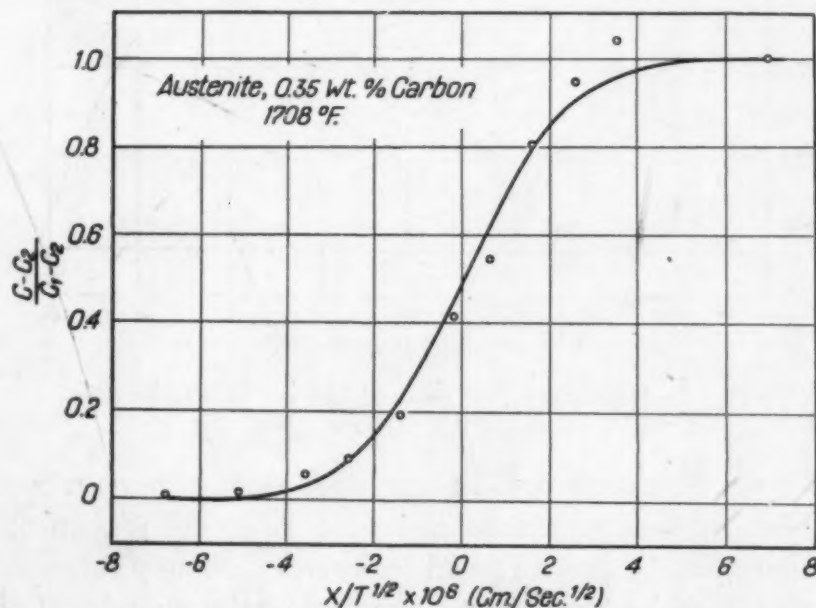


Fig. 4—Diffusion Curve for Molybdenum in Austenite. Experiment 10.

tance (δ) and the number of times (d) an atom moves from one position to the next per second (8), (9). If δ is taken as equal or proportional to the lattice parameter (a_0), it is obvious that if d is constant, D will vary as the square of the lattice parameter. Thus D cannot be independent of concentration. It was found (6) that a_0 increased linearly from 2.86063 Å for pure iron to 2.87261 Å for a 3.61 atomic per cent (6.05 weight-per cent) molybdenum alloy.

These figures can be used to show that D would be less than one per cent greater in the 6 per cent alloy than in pure iron if its change arose only from a change in δ . Since the present data indicate that the effect of concentration on D is small, it appears that the quantity d is also little affected by the molybdenum concentration.

It is possible that, as in the case of the diffusion of nickel and manganese (3), (4), D for molybdenum would increase if higher concentrations (such as 25 weight-per cent) were used. The D values for nickel and manganese increase far more rapidly than the

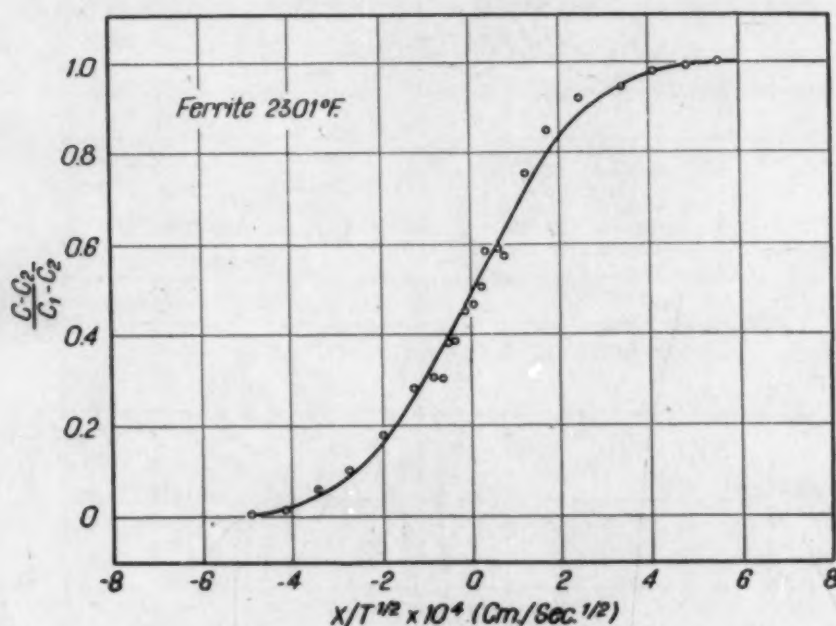


Fig. 5—Diffusion Curve for Molybdenum in Ferrite. Experiment 11.

change of jump distance would indicate if d were to remain constant.

Diffusion in binary systems involving large changes in a_0 with concentration must be accompanied by correspondingly large volume changes; that is, if both specimens were originally cylindrical and of the same diameter, after diffusion they would be expanded in diameter and in length where the concentration changes were such as to increase a_0 and contracted where a_0 decreased. If the increase is found to be greater than that predicted by the change in a_0 at some point n a_0 units from the interface (where n is an integer), it follows that the total number of atoms entering a slice $\Delta n a_0$ units thick at this point was greater than the total number of atoms leaving during diffusion and that one of the metals is diffusing more rapidly than the other. The shape change is therefore a test of the

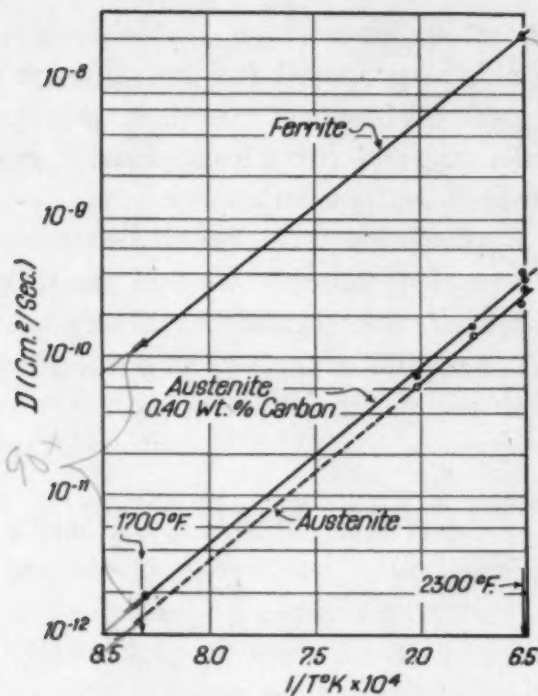


Fig. 6—Effect of Temperature on the Coefficient of Diffusion of Molybdenum in Austenite and in Ferrite.

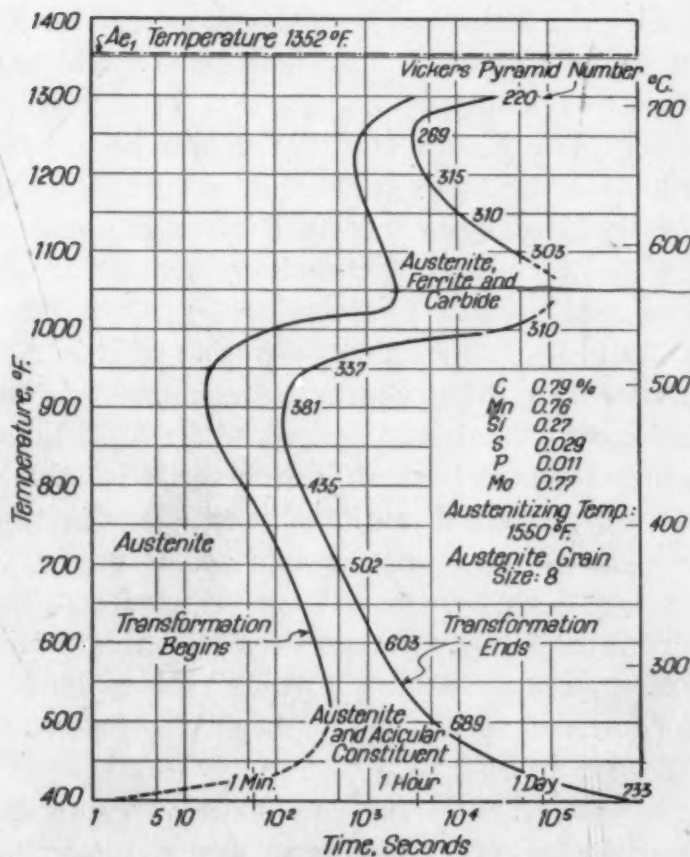


Fig. 7—S-Curve for 0.79 Per Cent Carbon Steel Containing 0.77 Per Cent Molybdenum.

assumption of "equal atom interchange", which seems to be necessary to several mechanisms proposed for the diffusion process.

From experimental evidence, Kirkendall (10) concludes that this assumption cannot be valid for the copper-zinc system and that zinc diffuses much more rapidly than copper in copper-zinc alloys.

The coefficient of diffusion as ordinarily measured must be a composite quantity dependent in some way on the properties of the individual metals involved, that is, on the ability of each to flow through the alloy. It is possible that the real nature of the composite quantity could be deduced if the following data were obtained concerning a pair of metals which form a continuous series of solid solutions:

The value of the ordinary diffusion coefficient D at all concentrations.

The self-diffusion coefficients of both pure metals.

The self-diffusion coefficients of each metal in the chemically homogeneous alloy at all concentrations.

Johnson (7) measured these quantities in the gold-silver system, but only at concentrations of 0, 50 and 100 atomic per cent. The coefficient of self-diffusion of pure gold was uncertain. More data on this system, say at 25 and 75 atomic per cent, would be extremely welcome.

Elkevich (11), in a general statistical treatment of substitutional diffusion in simple and body-centered cubic systems, also questions the validity of the assumption of equal atom interchange, and in order to account for variations of D with concentration, had to abandon it. He developed equations expressing D_{AB} , "the rate of diffusion of A in B," and D_{BA} , "the rate of diffusion of B in A," in terms of concentration. The difference between these was termed the "relative diffusion rate" and was designated by D_1 . In testing the ability of these equations to account for the variation of D calculated by the Matano method from data of Grube and Jedelev on the copper-nickel system, Elkevich employed the expression for D_{NiCu} and concluded that the equations were approximately correct. The reasoning is not clear since, if the expression for D_{CuNi} had been used the results would have been different. Further explanation of these results is therefore needed and indeed might prove very fruitful in explaining the diffusion process.

The foregoing discussion suggests some very attractive problems in mathematics as well as in experimental research. An ex-

ample of the former is the derivation and solution of a diffusion equation which applies to substitutional diffusion involving large changes in lattice parameter. After this problem has been solved, some interesting experiments could be performed to determine, by observation of shape or volume changes occurring during diffusion, what differences—if any—exist between rate of transfer of the various atoms involved. The radioactive tracer method seems to be the most promising alternative for attacking this problem. At present theoretical reasoning such as that of Elkevich, although useful, involves too many questionable assumptions to be regarded as conclusive.

THE ROLE OF DIFFUSION IN THE TRANSFORMATION OF AUSTENITE

The present state of our knowledge of the effect of alloying elements on the rate of transformation of austenite was briefly reviewed in the introduction. It is now appropriate to consider the conditions imposed on the diffusion of molybdenum during transformation by the findings of Bowman and Parke (5), (6) concerning the partition of molybdenum between ferrite and carbide.

Since the carbides formed by the isothermal transformation of austenite at 1300 degrees Fahr. (700 degrees Cent.) were found to contain considerably more molybdenum than those formed by tempering martensite at this temperature, Bowman and Parke predicted that the rate of diffusion of molybdenum was less in ferrite than in austenite. The diffusion experiments just described, however, show conclusively that the coefficient of diffusion is not less but is much greater in ferrite than in austenite. Owing to the manner in which the diffusion coefficient is defined, the rate of diffusion or rate of delivery of molybdenum atoms to the carbide depends not only on the diffusion coefficient but also on the concentration gradient. The term "diffusion rate", implying the mass flux through a unit area, is incorrectly applied when it refers to the diffusion coefficient, which is defined as this rate per unit concentration gradient. The magnitude of the concentration gradient depends on the nature of the carbide; and, since the composition, lattice structure, and degree of dispersion of the carbides formed by isothermal transformation are entirely different from those of the carbide formed on tempering, the concentration gradient associated with the former obviously may be quite different from that of the latter. The carbides formed from austenite at 1300 degrees Fahr. (700 degrees Cent.) in eutectoid iron-

carbon-molybdenum alloys were found to be of the complex face-centered type when more than 0.50 weight-per cent molybdenum was present (6).

A discussion of the relationship between the rate of growth of carbides containing molybdenum and the concentration gradient and diffusion coefficient of molybdenum in austenite is necessary to the explanation of the effect of molybdenum on the rate of transformation of austenite. The discussion, as given here, applies only to the rate of growth of isolated carbide particles or the rate of edgewise extension of pearlite.

The conditions prevailing during an early stage of the growth of carbide by isothermal transformation of austenite in a eutectoid steel differ in no way from those prevailing at some later period in the growth process; therefore we may assume that the carbides are invariant in composition during formation. After formation these carbides may, to be sure, change their composition by interchange of atoms with the ferrite phase which may be simultaneously precipitating, as in the formation of pearlite. This interchange, however, will not affect their rate of growth from austenite. In order to grow and maintain a definite composition, the carbide must acquire the various atoms involved at rates whose relative magnitudes are determined only by the composition of the carbide. The rate of growth of the carbide would be limited and determined by the slowest rate involved in the delivery of the various atoms to the carbide. If the carbide contained a sufficient quantity of molybdenum, its rate of growth would depend on the rate of diffusion of molybdenum, which depends on the coefficient of diffusion and the concentration gradient. Little or no diffusion of iron is necessary, since its concentration is but slightly greater in the austenite than in the carbide.

Obviously, during growth the rate of delivery of molybdenum atoms to the phase boundary must equal the rate at which they are transferred across the phase boundary. In order to see how this equality is maintained, let us define a quantity c_0 as the concentration of molybdenum in the austenite an infinitesimal distance from the growing carbide phase. The molybdenum concentration gradient $\left(\frac{\partial c}{\partial x}\right)_{c_0}$ at this point will then be uniquely determined by c_0 . The rate of flow of molybdenum atoms at this point is given by $D \left(\frac{\partial c}{\partial x}\right)_{c_0}$ as assumed in the derivation of the diffusion equation. As c_0 ap-

proaches zero the rate of flow increases but approaches a constant finite value, since the flow through the face of a semi-infinite bar does not become infinite when c_0 (the concentration of the face) approaches zero. However, as c_0 approaches zero the rate of transfer across the phase boundary does approach zero and probably in a linear manner.

If c_0 were equal to the original concentration (c_a) of the austenite, as it must be immediately after nucleation, there could be no concentration gradient; so, in order to perpetuate growth, c_0 must drop very rapidly to set up the concentration gradient necessary to deliver the molybdenum atoms at the same rate as they cross the boundary. (5)

If c_0 must become very small compared to c_a in order to decrease the rate of transfer and increase the rate of diffusion until they are equal, it can be said that the rate of growth is governed almost entirely by the diffusion coefficient only, since the concentration gradient at c_0 , and therefore the flow of molybdenum atoms, cannot be increased appreciably by further decreasing c_0 , that is, by increasing the rate of transfer across the boundary. (5)

If c_0 maintains some value intermediate between zero and c_a , the rate of growth will depend on the rate of transfer across the phase boundary.

An interesting example of a case where the rate of growth depends on the rate of transfer across the phase boundary rather than upon the coefficient of diffusion of the element involved is the growth of pearlite in eutectoid plain carbon steel. According to experimental work reported by Mehl (14), the rate of growth of a pearlite nodule increases as the temperature is lowered. The rate of growth cannot be governed by the coefficient of diffusion of carbon in austenite, since this coefficient decreases as the temperature is lowered. (6) The fact that the rate of growth is increased by a decrease in temperature proves that the carbon concentration gradient could not have reached its maximum value at the higher temperature involved. The maximum rate of transformation, then, is likely to occur at the temperature at which the maximum carbon concentration gradient is reached. It is unlikely that this condition coincides with the "nose" of the S-curve. Thus, because of the intrusion of a new process of nucleation at the "nose" of the S-curve, the rate of transformation of austenite to pearlite fails to reach the theoretical maximum. (7)

It is probable that c_0 is small compared to c_a during the growth (8)

of the complex molybdenum carbide in austenite and that the diffusion coefficient of molybdenum in austenite determines the rate of growth.

5K1P (7) The best evidence of this is that in order to account for the shape of the S-curve of a eutectoid molybdenum steel the rate of growth must decrease as temperature decreases in the temperature range where the complex carbide is formed. As the temperature drops, the coefficient of diffusion is known to decrease rapidly, but the rate of transfer across the phase boundary would probably increase, since the temperature is departing from that at which the rate of transfer is zero, that is, the A_{e1} temperature.

The sidewise growth of pearlite is probably also dependent on the coefficient of diffusion, but in this case the diffusion process is periodically interrupted by the formation of ferrite and therefore is a series of transient diffusion phenomena rather than a steady-state process such as is postulated for edgewise extension.

(10) The importance of the discovery that the coefficient of diffusion of molybdenum is much greater in ferrite than in austenite lies in the fact that it forms the basis for a possible explanation of the effect of molybdenum on the shape of the S-curve. For example, let us consider the S-curve of an 0.80 per cent carbon, 0.75 per cent molybdenum steel presented by Blanchard, Parke, and Herzig (12). (Fig. 7.) If the time interval between the start and end of transformation is used as a measure of the rate of transformation and if the rate is considered at successively lower temperatures starting from the A_{e1} temperature, it can be seen that the rate of transformation is slow in the pearlite region just below the A_{e1} temperature, that it increases to a maximum at about 1250 degrees Fahr. (675 degrees Cent.), decreases to a minimum at about 1050 degrees Fahr. (575 degrees Cent.), and finally increases very rapidly in the bainite region between 1050 and 900 degrees Fahr. (575 and 485 degrees Cent.). If it may be assumed that all reaction products above 1050 degrees Fahr. (575 degrees Cent.) are nucleated by carbide, as is pearlite (13), it is obvious that since the molybdenum content of these carbides was found to be high, the rate of their growth must depend on the coefficient of diffusion of molybdenum in austenite. In the temperature range between the A_{e1} and 1050 degrees Fahr. (575 degrees Cent.) the coefficient of diffusion of molybdenum decreases rapidly as the temperature decreases, but the rate of formation of carbide nuclei increases rapidly (14). The resultant effect of temperature

on these two quantities must be such as to cause the observed maximum in the transformation rate at 1250 degrees Fahr. (675 degrees Cent.).

This is consistent with the following qualitative consideration. If the overall rate depends primarily on, and varies directly with, two other variables, that is, the rate of nucleation and the coefficient of diffusion, the necessary condition for the occurrence of a maximum in the overall rate at an intermediate temperature is that a drop in temperature increases one of these variables (the rate of nucleation) and decreases the other (the coefficient of diffusion). If they were both increased or both decreased by a drop in temperature, at all temperatures involved, a reversal in the overall rate certainly could not occur.

In plain carbon steels a maximum does not occur at 1250 degrees Fahr. In fact Mehl (14) has shown that the rate of growth of a pearlite nodule in this temperature range actually increases as the temperature decreases. The rate of growth in this case does not involve the diffusion of molybdenum but only the diffusion of carbon. The coefficient of diffusion of carbon, however, decreases much less rapidly than that of molybdenum as the temperature drops. It seems that the concentration gradient of carbon must increase sufficiently rapidly with a decrease in temperature to overcome the decrease in the diffusion coefficient and to account for the observed increase in rate of growth. This may be pictured as due to the increasing stability of the carbide and the decreasing interlamellar spacing of the pearlite. Apparently any increase in the concentration gradient of molybdenum which may occur as the temperature is lowered is not sufficient to overcome the attendant decrease in the coefficient of diffusion. This is consistent with definite experimental evidence that the interlamellar spacing of pearlite formed at 1200 degrees Fahr. (650 degrees Cent.) is greatly increased by molybdenum, whereas the spacing of that formed at 1300 degrees Fahr. (700 degrees Cent.) is not noticeably changed (12).

A maximum does occur, of course, in the rate of transformation of plain carbon steels but only when the temperature has dropped to about 1000 degrees Fahr. (540 degrees Cent.). At this point the type of nucleation and therefore the nature of the transformation itself changes, as will be shown later.

At any given temperature the rate of formation of the nuclei

of the face-centered cubic carbide containing molybdenum is likely to be slower than the rate of formation of the nuclei of ordinary cementite, owing to the greater complexity and size of the unit cell (5), (6). This, together with the fact that the coefficient of diffusion of molybdenum rather than of carbon governs the rate of growth of the complex carbide, explains the general shift of the S-curve toward longer times or slower rates caused by molybdenum in the temperature range where the complex carbide is formed.

The products of transformation below 1050 degrees Fahr. (575 degrees Cent.) in the case under consideration are believed to be nucleated by ferrite (13). This means that the process is initiated by the appearance of ordinary ferrite and that the carbide (or other phase whatever its nature) is nucleated or at least makes its appearance as a result of, and therefore after, a certain increase in the carbon concentration of the surrounding austenite. Unless this ferrite is simply a high temperature martensite, which is not considered likely, its formation must be accompanied by the accumulation of carbon in the surrounding austenite.

That molybdenum is simultaneously rejected from the ferrite does not seem likely, for the iron-molybdenum equilibrium diagram shows that the addition of sufficient amounts of molybdenum to austenite leads to the formation of ferrite. If subsequent diffusion of molybdenum through the ferrite to the other phase is necessary to the continuation of the process, it would not be expected to retard the rate of the process nearly so much as at temperatures just above 1050 degrees Fahr. (575 degrees Cent.) where the diffusion of molybdenum in austenite was involved, for the coefficient of diffusion of molybdenum in ferrite is much greater than that in austenite. As the temperature is lowered from 1050 degrees Fahr. (575 degrees Cent.) the rate of the austenite transformation increases sharply and again reaches a maximum in the vicinity of 900 degrees Fahr. (485 degrees Cent.) and then decreases as the temperature is decreased further. This maximum is also the result of opposing effects of temperature on the two controlling variables (rate of nucleation and coefficient of diffusion), analogous to that described above as producing the maximum at 1250 degrees Fahr. (675 degrees Cent.) but involving in this case the formation of ferrite nuclei and the diffusion of carbon in austenite and possibly of molybdenum in ferrite.

Molybdenum is much less effective in retarding transformation at these lower temperatures; therefore it must be assumed that the

amount of diffusion of molybdenum necessary for transformation decreases rapidly as the temperature drops below 900 degrees Fahr. (485 degrees Cent.). This is in agreement with the conclusions of Fischer and Rose (15), who determined by magnetic measurements that the amount of "carbidegenic" elements such as chromium or molybdenum in the carbide phase is less in bainite than in pearlite.

✓ From these considerations it may be postulated that in an 0.80 per cent carbon, 0.75 per cent molybdenum steel:

(A) The observed maximum in the rate of transformation of austenite at 1250 degrees Fahr. (675 degrees Cent.) is the result of an increase in the rate of formation of carbide nuclei and a simultaneous decrease in the coefficient of diffusion of molybdenum in austenite as the temperature is lowered.

(B) The bay in the S-curve at 1050 degrees Fahr. (575 degrees Cent.) results from the fact that 1050 degrees Fahr. is the minimum temperature at which the probability of formation of carbide nuclei is greater than that of the formation of ferrite nuclei.

(C) The abrupt increase in transformation rate occurring when the temperature drops from 1050 to 900 degrees Fahr. (575 to 485 degrees Cent.) is due to a sudden change from nucleation by carbide to nucleation by ferrite and the resultant changes in the diffusion processes necessary for transformation.

(D) The observed maximum in the rate of transformation at 900 degrees Fahr. (485 degrees Cent.) is the result of an increase in rate of formation of ferrite nuclei and a simultaneous decrease in the coefficient of diffusion of carbon in austenite and possibly of molybdenum in ferrite as the temperature is lowered.

✓ This explanation of the effect of molybdenum and temperature on the rate of transformation of austenite agrees essentially with that offered by Blanchard, Parke, and Herzig (12), who ascribed the bay in the S-curve, or minimum rate occurring at 1050 degrees Fahr. (575 degrees Cent.), to the greater effect of molybdenum on the rate of formation of pearlite than on the rate of formation of bainite and to the fact that the effect of molybdenum on the former increased but the effect of molybdenum on the latter decreased with drop in temperature. In the 0.80 per cent carbon, 0.75 per cent molybdenum steel the temperature range in which both processes can occur simultaneously is apparently very narrow. The present explanation may be regarded as an extension of this line of reasoning by way of accounting for the effect of temperature and molybdenum

content on the rate of transformation by a consideration of the fundamental processes of nucleation and diffusion.

As it stands at present, our picture of the mechanism by which molybdenum affects the shape and position of the S-curve of a eutectoid steel may be summarized as follows: The necessity for the diffusion of molybdenum in austenite during the transformation of a molybdenum steel from austenite to pearlite causes the rate maximum observed at about 1250 degrees Fahr. (675 degrees Cent.). The general shift of the S-curve to longer times (slower rates) in the pearlite region caused by molybdenum results from the dependence of the rate of growth of the carbides on the coefficient of diffusion of molybdenum in austenite and a decrease in the rate of formation of carbide nuclei as their molybdenum content increases. The effect of molybdenum on the shape and position of the S-curve in the bainite region (below 1050 degrees Fahr.) is small since molybdenum does not greatly affect the rate of formation of ferrite nuclei and less diffusion of molybdenum is necessary to transformation.

THE EFFECT OF MOLYBDENUM ON THE RATE OF TRANSFORMATION OF AUSTENITE COMPARED TO THAT OF MANGANESE AND NICKEL

Values of the diffusion coefficients for molybdenum, manganese, and nickel at subcritical temperatures in austenite containing 0.80 per cent carbon and 0.50 per cent of the element under consideration have been estimated from the data presented here and from the data of Wells and Mehl (3), (4). The relative magnitudes of the D values for molybdenum, manganese, and nickel are, respectively, 9, 4.4, and 1 at 1050 degrees Fahr. (575 degrees Cent.) and 4, 3, and 1 at 1500 degrees Fahr. (820 degrees Cent.).

Manganese and nickel also decrease the rate of transformation of austenite in the pearlite region but to a less degree than molybdenum, nickel being the least effective of these when present in equal weight or atomic proportions. The relative effectiveness of these elements is therefore not that to be expected from a consideration of their relative coefficients of diffusion alone. The importance of the coefficient of diffusion of these elements in determining the transformation rate depends on the quantity of these elements which must diffuse to the carbide phase during the growth of the carbides. In the case of nickel this quantity is thought to be small (16) (21), but it is known that considerable amounts of manganese can occur in the carbide phase (25), (26).

Quantitative data on the partition of nickel and manganese between the carbide and ferrite phases during isothermal transformation of austenite are needed to explain fully the effects of these elements. As suggested in the discussion of molybdenum carbides, the rate of formation of carbide nuclei also would be expected to depend on their manganese or nickel content. It is possible that elements which do not form a part of the carbide phase influence the rate of formation and growth of cementite nuclei since their concentration near the interface in the surrounding phase would increase as the carbide grew by the acquisition of iron and carbon atoms. Owing to the initial predominance of the iron atoms and the small effect of alloying elements on the rate of diffusion of carbon in austenite, we would not expect the rate of formation or the rate of growth to be as greatly affected by such elements as by molybdenum.

It is believed that the mechanism by which manganese and nickel induce hardenability is fundamentally different in at least one respect from that proposed for molybdenum. Examination of the appropriate equilibrium diagrams (17), (18), (19) reveals that the addition of sufficient molybdenum to iron produces the alpha phase at temperatures where only the gamma phase of pure iron is stable, but the addition of sufficient nickel or manganese to carbon-free iron produces the gamma phase at temperatures where only the alpha phase of pure iron is stable. This amounts to the statement that molybdenum raises the A_{e_3} critical temperature whereas manganese and nickel lower it. In either case the critical point is no longer sharply defined but represents a range of temperatures in which the alpha and gamma phases exist simultaneously in equilibrium as required by the phase rule. The upper and lower temperature limits of this two-phase region are fairly well established in the iron-molybdenum system, since no great hysteresis is associated with the change in phase; but in the iron-nickel and iron-manganese systems these limits are usually represented only by broad temperature ranges, since considerable time is required for the phase changes to take place at subcritical temperatures in these systems. (Recently these limits in the iron-manganese system have been determined with some precision (27). Reaction periods as great as two years were used in some cases to approach equilibrium.) This is clearly illustrated by the dilatometric measurements of Scheil (17) of the isothermal rates of transformation of austenite to ferrite at subcritical temperatures in carbon-free alloys of iron and nickel (4.8 weight-

per cent nickel). This deliberate behavior of the gamma-to-alpha transformation is not limited to alloys of iron with elements which lower the A_3 temperature, as shown by the work of Austin and Pierce (24) on iron-chromium alloys. The time associated with the transformation of iron-chromium alloys, however, is much less than that for iron-manganese and iron-nickel alloys.

The important point is that the presence of carbon is not necessary to the retardation of the rate of transformation of austenite to ferrite and that manganese and nickel would be expected to retard this rate simply by their presence in the original austenite and in the ferrite resulting from transformation, even if they had no effect on the rate of formation or growth of carbide nuclei.

The magnitude of the effect of nickel or manganese on the rate of gamma-to-alpha transformation, in alloys containing carbon, would depend, of course, on the amount of carbon present (20) and in some way on the relative mobility of the manganese or nickel and iron atoms. This mobility is probably related to the rates of diffusion of these elements in austenite and in ferrite, but in a manner not clear at present. Nickel, even at concentrations as high as four per cent, simply shifts the S-curve to the right (16) at all temperatures between the A_{e1} and the A_r'' and produces no bay in the S-curve. This supports the conclusion that the mechanism by which nickel retards the transformation rate is fundamentally simpler than that proposed for molybdenum. Nickel has less effect on the rates of nucleation and growth of the carbide phase and therefore causes no reversal in the rate of transformation in the pearlite region. The inherent sluggishness of the gamma-to-alpha transformation in iron-nickel alloys, together with the lowering of the critical temperature brought about by nickel, would account for the general shift of the S-curve to the right.

As pointed out by Austin (23) and Mehl (22), the instability of the austenite lattice depends on some power of the difference between the equilibrium temperature and the existing temperature rather than on the existing temperature alone. Therefore nickel and manganese would be expected to increase the stability of the austenite lattice at any given temperature if only by virtue of their ability to lower the equilibrium temperature.

It is possible that manganese promotes hardenability by a combination of the mechanisms proposed for molybdenum and nickel.

USE OF DIFFUSION DATA IN COMPARING HOMOGENIZATION RATES

Without defining the degree of heterogeneity except to say that it is uniquely determined by existing concentration gradients which change with time according to the diffusion equation, we may develop two important relationships giving the time necessary to produce a given change in heterogeneity in terms of the temperature, where only one diffusing element is being considered, and in terms of Q and A in the equation $D = Ae^{-Q/RT}$, when comparing the rates of homogenization of two different elements at the same temperature.

It can be shown mathematically that even if the initial concentration is a very complicated function of distance, any concentration change at a given point in the steel and therefore any change in the concentration gradient is determined uniquely by the product of the diffusion coefficient (D) and the time (t). (We are considering a solid solution which is inhomogeneous but not saturated with respect to the diffusing element.)

Expressing D as $Ae^{-Q/RT}$, we see that if a certain change in heterogeneity can be obtained in a time (t_1) when A , Q , and T have values A_1 , Q_1 , and T_1 , then the time t_2 necessary to obtain the same improvement with another set of values A_2 , Q_2 , and T_2 is given by

$$\frac{t_2}{t_1} = \frac{A_1 e^{-Q_1/RT_1}}{A_2 e^{-Q_2/RT_2}}$$

The time t_2 to obtain a certain degree of improvement in homogeneity at a temperature T_2 in terms of the time t_1 required to obtain this same improvement at temperature T_1 when A and Q are the same; that is, for a given diffusing element, is given by

$$\frac{t_2}{t_1} = e^{\frac{Q}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)} \quad [1]$$

The time t_2 at a given temperature T_1 necessary to obtain a given degree of improvement with respect to an element with diffusion constants A_2 and Q_2 in terms of the time t_1 necessary for the same degree of improvement with respect to an element with diffusion constants A_1 and Q_1 is given by

$$\frac{t_2}{t_1} = \frac{A_1}{A_2} e^{(Q_2 - Q_1)/RT} \quad [2]$$

Applying [1] to the diffusion of carbon in austenite ($A = 0.22$, $Q = 34,200$, in the presence of 0.80 per cent molybdenum and 0.40

per cent carbon) (1), we find that if $T_1 = 2000$ degrees Fahr. (1095 degrees Cent.) and $T_2 = 1800$ degrees Fahr. (980 degrees Cent.), then $t_2/t_1 = 3.05$. In other words, at 2000 degrees Fahr. (1095 degrees Cent.) we can accomplish a given result in about a third of the time necessary at 1800 degrees Fahr. (980 degrees Cent.).

For homogenization with respect to molybdenum at these temperatures ($A = 0.091$ and $Q = 59,000$, in the presence of 0.40 per cent carbon), $t_2/t_1 = 6.87$ and the time would be reduced by a factor of $1/6.87$ by the 200-degree increase in temperature.

Applying [2] to compare carbon and molybdenum at 1800 degrees Fahr. (980 degrees Cent.), $t_2/t_1 = 49,000$; so that it would take 49,000 times as long to homogenize with respect to molybdenum as with respect to carbon, assuming the same degree of inhomogeneity at the start.

Using [2] to compare the rate of homogenization of molybdenum in pure ferrite ($A_2 = 3.467$, $Q_2 = 57,700$) and in pure austenite ($A_1 = 0.068$, $Q_1 = 59,000$) at 1800 degrees Fahr. (980 degrees Cent.), we find that $t_2/t_1 = 86$. This factor is essentially independent of temperature, owing to the similarity of the Q values. Complete homogenization with respect to an element such as molybdenum cannot, of course, be realized in steels at subcritical temperatures, since molybdenum segregates to the precipitated carbide phase. It is possible that concentration gradients covering relatively large distances such as those existing in a banded structure could be lowered somewhat at subcritical temperatures by passage of molybdenum through the ferrite from carbides rich in molybdenum to those containing less molybdenum. However, it is very probable that elements such as nickel, which are thought to remain to a large extent in the ferrite (16), (21), also diffuse more rapidly in ferrite than in austenite. If this is the case it is reasonable to suppose that steel can be homogenized with respect to these elements more rapidly just below the critical temperature than above it, especially if the carbon content is low, so that the carbon particles do not obstruct appreciably the diffusion process.

In very low carbon iron-molybdenum alloys, the homogenization process is unquestionably much more rapid just below the critical temperature than just above. According to Fig. 6, the rate of homogenization (diffusion) in an alloy of pure iron plus 2.0 weight-per cent molybdenum in ferrite just below the critical temperature (1700 degrees Fahr.; 925 degrees Cent.) is about the same as that in aus-

tenite 500 degrees Fahr. above the critical temperature (2200 degrees Fahr.; 1200 degrees Cent.).

SUMMARY

The diffusion coefficients of molybdenum in pure austenitic and ferritic iron-molybdenum alloys and in pure austenitic iron-molybdenum-carbon alloys containing 0.40 per cent carbon have been measured. The diffusion coefficient of molybdenum in ferrite was found to be much greater than that in austenite. The presence of 0.40 per cent carbon was found to increase slightly the coefficient of diffusion of molybdenum in austenite. These coefficients are given by the following equations:

Molybdenum in Austenite	$D_A = 0.068 e^{-59,000/RT}$
Molybdenum in Austenite Containing 0.40 Weight-Per Cent Carbon	$D_{Ac} = 0.091 e^{-59,000/RT}$

Molybdenum in Ferrite	$D_F = 3.467 e^{-57,700/RT}$
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The composite nature of the coefficients of diffusion of binary substitutional solid solutions has been discussed.

A mechanism has been proposed to explain the effect of molybdenum on the shape of the S-curve on the basis of nucleation rates, diffusion rates, and partition data. The rate maxima in the S-curve of an 0.80 per cent carbon, 0.75 per cent molybdenum steel are accounted for by the increase in the nucleation rates and the simultaneous decrease in diffusion rates accompanying a drop in temperature. The abrupt increase in transformation rate occurring as the temperature drops from 1050 to 900 degrees Fahr. (575 to 485 degrees Cent.) is accounted for by an abrupt change in the type of nucleation occurring.

The mechanism by which manganese and nickel alter the shape and position of the S-curve may be fundamentally different from that proposed for molybdenum.

Equations expressing relative rates of homogenization in terms of diffusion coefficients have been presented.

ACKNOWLEDGMENT

The author wishes to acknowledge the valuable contributions of the various members of the staff of the research laboratory of Climax Molybdenum Company who ably assisted both in the experimental work and in the preparation of this paper.

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DISCUSSION

Written Discussion: By E. P. Klier, instructor in metallurgy, Pennsylvania State College, State College, Pa.

Numerous investigators of the phenomena associated with austenite decomposition have proposed mechanisms whereby such decomposition occurs. Few, however, have presented such detailed outlines of their concepts as is given in this paper.

In this consideration of austenite decomposition, certain fundamental notions are involved. Without regard to their relative importance the more important premises may be considered as:

1. Diffusion data, obtained in the conventional manner, are indicative, at least qualitatively, of the conditions under which diffusion takes place during the decomposition of austenite. This then demands that all rates of diffusion decrease with decreasing temperature.
2. Pearlite is nucleated by carbide.
3. Bainite is nucleated by ferrite.
4. Such nucleation as is provided for in 2 and 3 is subject to no modification as has been found necessary in treating certain of the age hardening systems—notably copper-aluminum—to account for recovery.

Experimental evidence such as to invalidate points 1 and 4 has recently been reported by Klier and Lyman.

The 3 per cent chromium-0.40 per cent carbon steel studied by Klier and Lyman has an S-curve as presented in Fig. A. Consider the transformation characteristics at 500 degrees Cent. (930 degrees Fahr.).

Precipitation of a carbon-poor structure begins roughly in one hour. After about one day at temperature, a second reaction product becomes evident. The two reactions progress together for an unstated time.

An X-ray analysis of the specimens reacted at this temperature indicates the following conditions as obtaining:

1. After 26 hours at temperature there is present austenite of greater than 0.38 per cent carbon content (the precise carbon content of this aus-

tenite has not been determined, but is estimated to be about 0.60 per cent).

2. This high-carbon austenite is present at 67.5 hours.
3. After 120 hours, evidence of the carbon-rich austenite is no longer present, the indication being that all the austenite is of 0.38 per cent carbon content.

Thus between 26 hours and 67.5 hours there is present in the steel a low-carbon structure, which for convenience has been called ferrite, an austenite containing 0.38 per cent carbon and an austenite containing an estimated 0.60

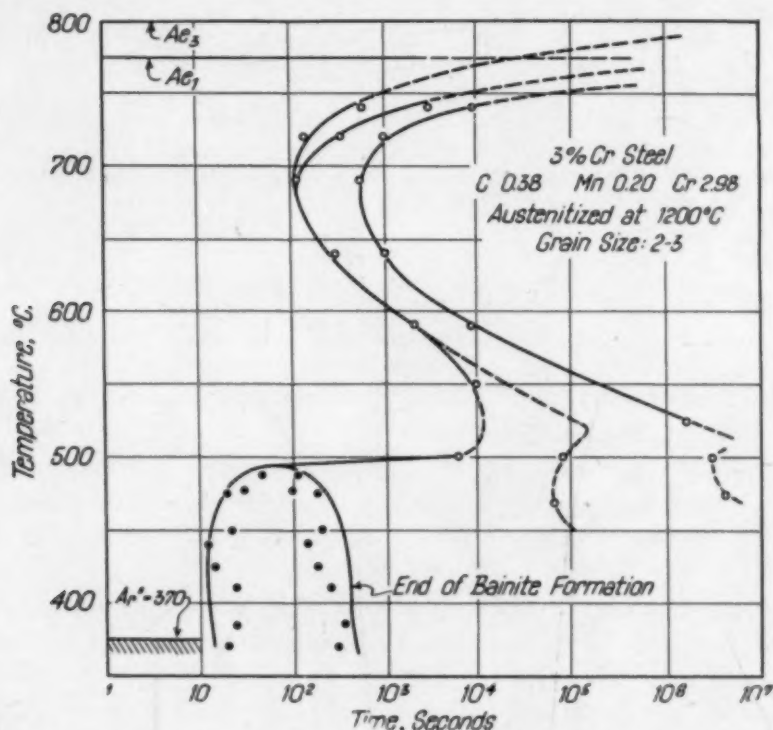


Fig. A—Isothermal Transformation Diagram for 3 Per Cent Chromium Steel. Open Circles, Points Determined Metallographically. Solid Circles, 0.5 Per Cent and 99.5 Per Cent Extension in Dilatometer.

per cent carbon. From metallographic examination of specimens it was decided that the enriched austenite surrounds the ferrite.

The carbon gradient surrounding each ferrite needle must be somewhat as represented in Fig. B. The ordinate is carbon content—the abscissa is the distance removed from the center of the ferrite needle in some crystallographic direction. A represents approximately the interface.

Assume now that diffusion of carbon has taken place in the austenite only. It is immediately evident that the rate of diffusion has varied as a function of the composition.

The variation of the carbon diffusion is very interesting. To the left of the interface A carbon has diffused at a greater rate than it has in the region immediately to the right of the interface. Yet this carbon-rich zone has had to move a very great distance to account for its carbon content, as well as to allow the precipitate structure to develop to its final size.

It is important to note that the rate of carbon diffusion varies in the manner shown above under these conditions. Of still greater importance is the realization that this carbon diffusion has from the start been up a concentration gradient, rather than down as would normally be expected.

Of importance is the time required at this temperature for the development of this concentration gradient. This time may be considered as something over an hour. A specimen of this steel transformed at 475 degrees Cent. for 3 minutes is about 10 per cent transformation product. A consequence of this transformation is the enrichment of some austenite to a degree comparable with that

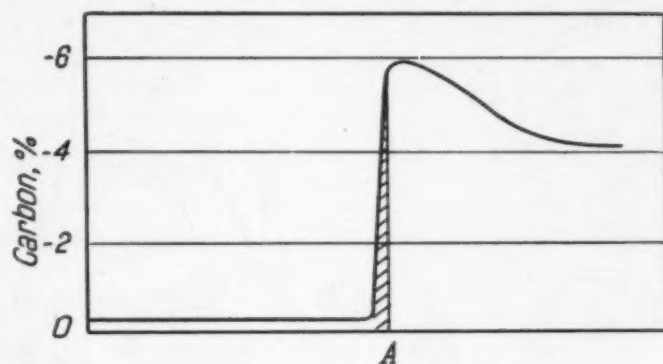


Fig. B

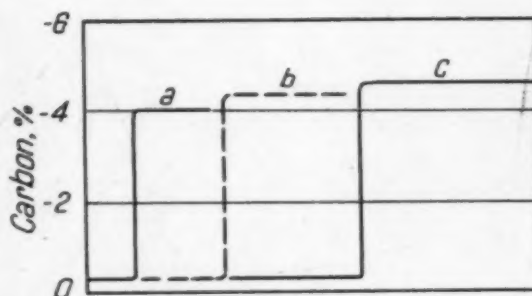


Fig. C

for the specimen transformed at 500 degrees Cent. (930 degrees Fahr.) for 26 hours. Transformation at this temperature begins in 30 seconds. Therefore, the apparent increase in the rate of carbon diffusion must be by a factor of the order of 120 times on lowering transformation temperature 25 degrees Cent.

Certain other data could be presented to show the same phenomena described above. It is necessary to conclude that the rate of carbon diffusion may increase with falling temperature and may be discontinuous at constant temperature. Diffusion data obtained by the normal methods thus are not adequate to help in the solution of the problem considered.

Klier and Lyman state "that concepts of nucleation and grain growth cannot assist in the formulation of a theory of the bainite reaction". This statement may be elucidated as follows:

In Fig. C are presented curves representing the carbon concentration measured along some crystallographic direction away from the center of a

nucleus at the instant of its formation, and after various degrees of its growth. Such transformation should be readily observable under the microscope and should develop a characteristic microstructure composed of a series of different sized precipitate particles—all otherwise metallographically identical.

One other condition only is realizable to account for this transformation on the assumption that the carbon diffusion takes place in the austenite lattice. This consists in the formation of the precipitate particle as low-carbon austenite

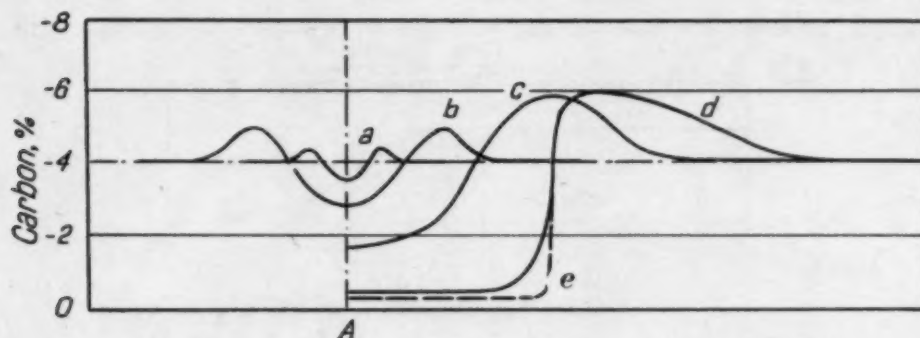


Fig. D

followed by a lattice transformation of this low-carbon area. These conditions are indicated in Fig. D.

A in Fig. D is selected as some point in the austenite matrix which at some future time will become the center of a ferrite needle. Curves a, b, c and d represent the carbon content in the face-centered cubic lattice after the indicated relative growth of the low-carbon region. Curve e is little different from curve d, the main difference being that the dotted line represents the region which has converted to the body-centered cubic lattice—the ferrite structure. This scheme calls for a steady increase in the diffusion coefficient with increasing transformation until a maximum is reached; this is followed by a large decrease—to account for the long delay in the erasure of the high-carbon areas.

If the second description of transformation to "ferrite" and bainite be considered correct, the metallographic evidence would be quite characteristic. Thus the initial stages of transformation would probably escape metallographic detection. This is especially true due to the occurrence of the martensite reaction on cooling of these steels. In the later stages of transformation, however, it should be possible to develop relief effects, between the very low carbon martensite and the relatively high carbon martensite surrounding it. Structures developed in various steels in this range are presented in Figs. E and F.

In Fig. E it is especially evident that two precipitate structures are developed. The first is crisply outlined resulting from the presence of well defined grain boundaries. The second structure is poorly defined at the boundaries evidently being etched into relief. On continued holding this second structure disappears with an increase in amount of the first considered precipitate. The small particles to be expected from nucleation and growth are not present in any of the structures examined.

It appears reasonable to accept the second description of the mechanism by which the ferrite considered and bainite are formed.

It is evident that the concept of nucleation and grain growth is not adequate to account for the phenomena which have been described. Two experimentally determined facts—the diffusion phenomena described above and the temperature dependence of the degree of completion of the bainite reaction (a point well brought out by numerous investigators) virtually preclude the possible modification of the nucleation and grain-growth theory to account for the bainite reaction.

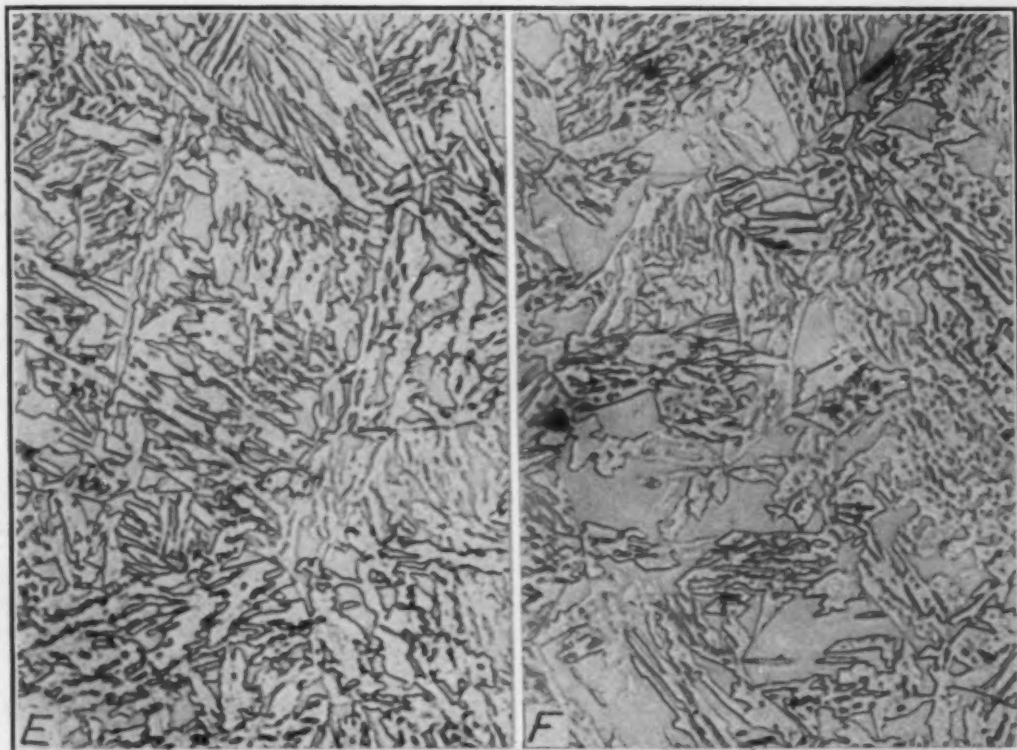


Fig. E—Silicon-Manganese-Molybdenum Steel Containing 0.51 Per Cent Carbon Transformed 300 Seconds at 970 Degrees Cent. Etched in Vilella's Reagent. $\times 1000$.

Fig. F—Manganese-Molybdenum Steel Containing 0.29 Per Cent Carbon Transformed 100 Seconds at 970 Degrees Cent. Etched in Vilella's Reagent. $\times 1000$.

The theory of nucleation and grain growth does not explain the martensite or bainite reactions. Even in an explanation of the pearlite reaction, serious difficulties are encountered. Only by allowing both the rates of nucleation and growth to vary can the known experimental data for steels be explained.

The transformation phenomena occurring in hypereutectoid steels, i.e., the proeutectoid precipitation of carbide, does not argue well for the notion that pearlite is nucleated by this carbide. It would seem that even here the concept of nucleation does not permit of simple interpretation. Further, in the hypoeutectoid steels, as is obvious from proeutectoid ferrite precipitation, the formation of a ferrite nucleus is much easier than the formation of a carbide nucleus. Thus in hypoeutectoid steels, ferrite nucleation does not restrict the pearlite reaction, while in hypereutectoid steels, carbide nucleation does not restrict this reaction.

It would appear that for the pearlite reaction the rates of nucleation of ferrite and carbide are interdependent and equal once proeutectoid precipitation is complete. In terms of nucleation, this virtually calls for a duplex nucleus, which though a rather improbable notion could at least account for the separation of the proeutectoid reaction from the pearlite reaction.

Written Discussion: By Howard S. Coleman, department of physics, The Pennsylvania State College, State College, Pa.

It occurs to me that the paper in question is very well done, and is of considerable importance from both the theoretical and experimental point of view. Insofar as the experimental results are concerned, it is evident that a great many useful data have been accumulated.

It occurs to me that several valuable theoretical points have been made. It would be very desirable to pursue Mr. Ham's reasoning a little further with respect to the number of "jumps" an atom takes per second as a function of the concentration, inasmuch as the results will be applicable to other similar metallic systems.

Author's Reply

I wish to thank Mr. Klier for his discussion of this paper. It provides an opportunity to emphasize and clarify some of the more fundamental concepts of the diffusion process. I do not suggest that diffusion data obtained in the conventional manner are indicative even qualitatively of the conditions under which diffusion takes place during the decomposition of austenite, nor that all rates of diffusion decrease with a decrease in temperature. The conditions under which diffusion takes place are determined by the type and rate of nucleation and by the nature of the products of transformation. I do assert that all diffusion coefficients decrease with a decrease in temperature and I have carefully distinguished between diffusion rates and diffusion coefficients in this paper. The diffusion rate increases as the concentration gradient increases. The diffusion coefficient does not. The diffusion rate can be increased by a drop in temperature only if the concentration gradient is thereby increased. It is not surprising that Mr. Klier has found evidence that diffusion rates can increase as temperature decreases. Studies of the rate of growth of pearlite in plain carbon steels confirm this. However, nowhere in the evidence cited by Mr. Klier do I find any indication that diffusion coefficients can increase as temperature decreases or that they are not applicable to the diffusion processes occurring during the transformation of austenite. Mr. Klier's statement that the rate of diffusion of carbon may be discontinuous at constant temperature is certainly correct. It is discontinuous for instance in the sidewise growth of pearlite. Fig. B presented by Mr. Klier showing the concentration of carbon as a function of the distance during the growth of ferrite seems quite reasonable qualitatively. It seems to me, however, to show clearly that carbon is diffusing in the austenite away from the advancing ferrite interface in the direction expected from the concentration gradient shown. In stating that carbon diffuses against the concentration gradient, perhaps Mr. Klier refers to the transfer of carbon atoms across the phase boundary, although I see no necessity for such transfer from ferrite to austenite, that is, against the so-called concentration gradient at the interface. Diffusion coefficients measured in the ordinary manner,

of course, give no indication of the rate at which the atoms involved might cross the phase boundary. Such processes and the concentration differences associated with them, if referred to as diffusion, should be carefully distinguished from the diffusion processes occurring in a single phase under the influence of ordinary concentration gradients.

Referring again to the growth of ferrite in Fig. B, Mr. Klier says that the rate of diffusion has varied with composition. The manner in which the diffusion coefficient of carbon in austenite varies with concentration is well known. Aside from this, the rate of diffusion can be different at different concentrations only if the concentration gradient also is different.

In view of what I have already said, I cannot agree with Mr. Klier concerning the nature of the diffusion processes which might accompany the formation of bainite. The assumption of the duplex ferrite-carbide nucleus seems to complicate the picture of the austenite-to-pearlite reaction unnecessarily since ferrite nucleation automatically follows the nucleation and growth of a carbide plate.

I wish to thank Dr. Coleman for his encouraging remarks. I certainly intend to pursue further the theory of substitutional diffusion, and any suggestions by Dr. Coleman would be very welcome indeed.

CAPILLARITY OF METALLIC SURFACES

BY E. R. PARKER AND R. SMOLUCHOWSKI

Abstract

Certain phenomena observed in brazing and coating operations can be roughly correlated on the basis of a simple consideration of surface energies and geometrical factors. It is shown that the balance of changes of the solid-liquid, solid-air and liquid-air surface energies depends upon the capillary roughness of the metallic surface. The roughness is approximated by a groove of an angle α inclined to the surface of the metal at an angle β .

Experiments were made with liquid silver spreading on variously treated iron (6 per cent Mo) surfaces at 1200 degrees Cent. (2190 degrees Fahr.). The dependence on the angle α was qualitatively checked on 60, 90 and 120-degree grooves machined in the surface. Furthermore, the influence of grinding, polishing and etching was also studied. Liquid metal spreads best on finely ground or on polished and etched surfaces both of which have fine capillary structure; it does not spread on polished surfaces under the same conditions.

THE spreading of liquids over the surface of solids is important in many engineering fields. In metallurgy this phenomenon is of particular interest because of problems arising in brazing and coating. For example, it is very difficult to get liquid silver to flow over a smooth, clean surface of iron. It will be shown later, however, that silver spreads very readily if that surface has had certain treatments. The considerations outlined herein are helpful in understanding some of the strange happenings reportedly occurring in brazing and coating operations.

The behavior of a liquid on a solid metallic surface depends entirely upon the condition of the surface. A critical survey of various theories of capillarity and of spreading of liquids on solids¹ indi-

¹R. S. Burdon, "Surface Tension and the Spreading of Liquids," Cambridge University Press, 1940.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. Of the authors, E. R. Parker is associated with the University of California, Berkeley, Cal., and Roman Smoluchowski is associated with the Research Laboratory, General Electric Co., Schenectady, N. Y. Manuscript received May 31, 1944.

cates that there is no simple and complete theory of the phenomena involved. Therefore, in the following attempt to correlate and to explain qualitatively certain observations made on the spreading of liquid metals on solid metals, considerations have been limited to the notion of surface energy and to some simple geometrical factors. No attempt has been made to go into the details of the atomic picture of the mechanism of the spreading of liquids.

THEORETICAL CONSIDERATIONS

Spreading of a liquid on a solid surface depends upon the relative magnitudes of the surface energies (per unit of area) of the air-solid surface γ_s , air-liquid surface γ_L and solid-liquid surface γ_{SL} .² Spreading of the liquid decreases the air-solid surface and increases the other two surfaces. If the balance of these energy changes is negative, that is, if the total energy of the system decreases, then the liquid will spread, otherwise it will tend to cover a minimum area of the solid. The following considerations deal only with small quantities of the liquid so that surface phenomena play the preponderant role, while gravity and hydrodynamics are of secondary importance. The condition for spreading is thus:

$$\gamma_s \delta s + \gamma_{SL} \delta_{SL} + \gamma_L \delta_L < 0 \dots\dots\dots (1)$$

where δ_s, δ_L and δ_{SL} are the changes of the area of the three surfaces. On a flat surface $\delta_L = \delta_{SL} = -\delta_s$ approximately, and the condition for spreading is:

$$\gamma_L + \gamma_{SL} - \gamma_s < 0 \dots\dots\dots (2)$$

If the surface of the metal is covered with grooves such as those schematically illustrated in Fig. 1, the energy balance is altered and the liquid will spread more readily over the surface of the solid. In Fig. 1 the angle of the grooves is α and the bisector of this angle makes an angle β with the direction normal to the surface of the liquid. If the distance between the edges of the groove is of capillary size and the angle α is not too large, then the liquid will rise to the top of the groove. The grooves in this case act like capillary tubes causing the liquid to move rapidly in them. Let us assume for sim-

²In these considerations it is also possible to use the surface tension which is numerically equal to surface energy.

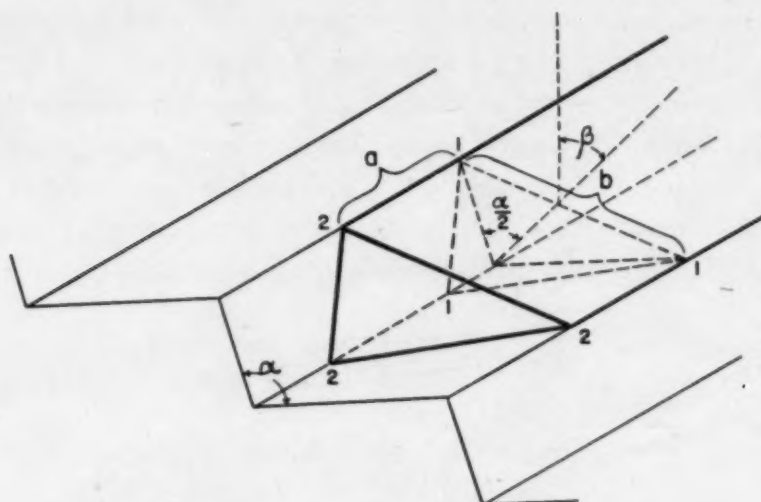


Fig. 1—Schematized Capillary Grooves on a Metallic Surface.

plicity that the front surface of the liquid is flat and that it has moved from position 1 to position 2. The following changes of area have occurred:

$$\delta_L = a b; \delta_S = -\delta_{SL} = -a b \cos \beta \csc \frac{\alpha}{2}$$

Thus the total energy has changed by an amount proportional to

$$A = \gamma_S - \gamma_L \sin \frac{\alpha}{2} \sec \beta - \gamma_{SL} \dots \dots \dots (3)$$

and the condition for spreading of the liquid is $A < 0$ which depends upon angles α and β . Thus on a flat, polished surface ($\alpha = 180$ degrees) the liquid may not spread at all ($A > 0$) but on a grooved or etched surface it will. Etching does not produce, in general, regular grooves on the metallic surface; however, the structure of an etched surface can be considered as made up of an irregular superposition of short grooves. The considerations remain valid. Since the surface energies depend on temperature, the condition $A < 0$ may be satisfied in one range of temperature and not satisfied in another.

Formula (3) helps also to understand the observed influence of the orientation of grains on spreading. Etching reagents preferentially expose certain crystal planes and thus the angle β changes from one grain to another. In this way the energy balance A is influenced by the orientation of the individual grain.

On the basis of these considerations, it seems reasonable that liquids should spread much better on rough (etched) metal surfaces (small α) and (depending upon the type of etching reagent used) spreading should be better on those grains of metal on which the exposed crystal planes are about equally inclined to the surface (small β). Similar considerations presumably govern the relative value of various etching reagents and various etching methods.

Whenever the grooves are too wide or too flat to be filled up to the edges by capillary action the liquid will move along the bottom of the groove and an estimate of the effect of the angle can be obtained in a different manner. It is a known fact that liquids rise between two vertical parallel plates by an amount which is approximately proportional to the reciprocal of the distance between the plates. Similarly, in the case of grooves on a metallic surface, the capillary action is strongest right on the bottom of the groove, and there the liquid may be expected to spread first, forming a layer on which subsequent layers of liquid move with greater ease. The volume of liquid, which is strongly affected by the proximity of the two walls of the groove, depends on the angle α between the walls.

For $0 < \alpha < \frac{\pi}{2}$ this volume is approximately proportional to $\frac{1}{\sin \alpha}$

and for $\frac{\pi}{2} < \alpha < \pi$ to $\text{Ctg } \frac{\alpha}{2}$. This consideration is important in the

case of large vertical grooves in which the range of capillary forces is relatively small so that gravity has to be taken into account. In this case the height to which the liquid will rise is not only affected by the balance of the surface energies but also by the volume of liquid which is to be raised.

EXPERIMENTAL RESULTS

Experiments were made on flat metallic surfaces subjected to various treatments and also on grooved surfaces in order to study the fundamental mechanism of spreading. The base metal was iron, to which about 6 weight per cent molybdenum was added to prevent the occurrence of α - γ transformation.

Fig. 2 illustrates the effect of the angle α on the rise of liquid in grooves. The iron sample had small V-shaped grooves 0.005 inch deep machined in the surface. Three angles were used: 60,

90, and 120 degrees. The iron sample was placed in a hydrogen furnace with the grooves vertical and with a little silver at its base. It was then heated at 1200 degrees Cent. (2190 degrees Fahr.) for 20 minutes. The silver melted and rose rapidly in the 60-degree grooves; slowly in the 90-degree grooves; and it hardly rose at all in the 120-degree grooves. This behavior is in agreement with the qualitative conclusions from the theoretical considerations.

A special study was made of the influence of various surface treatments of the iron samples. For this purpose an area of about

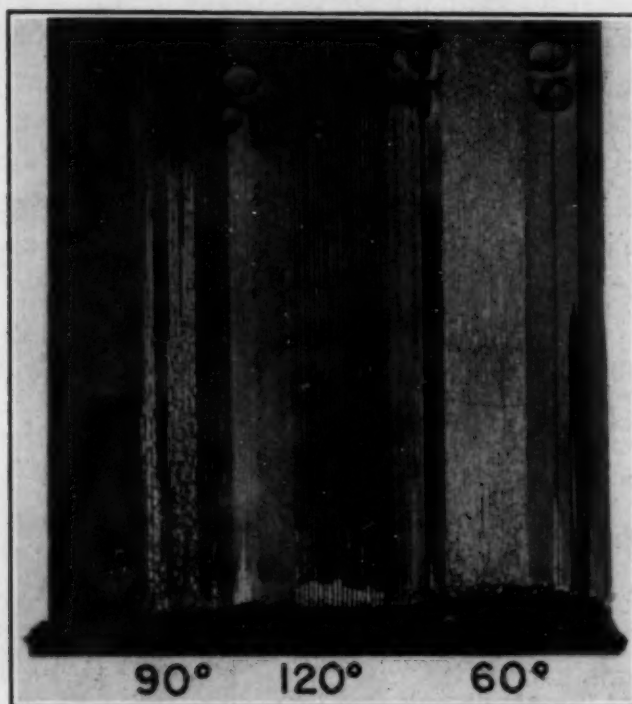


Fig. 2—Liquid Silver Rising on an Iron Surface Covered with Capillary Grooves of 60, 90 and 120 Degrees. (actual size)

5 square inches was treated in various ways, i. e., with a grit belt, sandpaper, polishing and etching with various reagents. By means of a profilometer the average³ roughness on the surface was determined. Then a small disk of silver sheet was placed in the center of each iron sample. The silver was melted in contact with the iron by heating in a hydrogen furnace at 1200 degrees Cent. (2190 degrees Fahr.) for about 20 minutes. The sample was cooled in hydrogen in the water-cooled chamber of the furnace. Fig. 3 shows a column of silver advancing in a groove ($\times 100$, No. 50-grit belt,

³Root mean square deviation from perfect flatness.

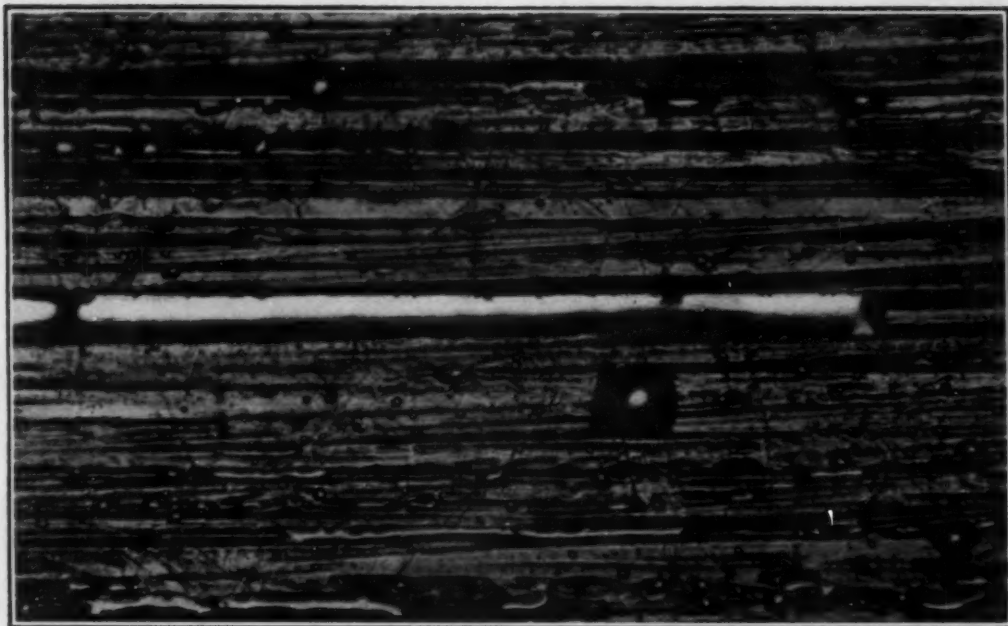


Fig. 3—Column of Silver Moving Forward in a Groove on a Ground Iron Surface (50-Grit Belt, Roughness 65×10^{-6} Inches, $\times 100$).

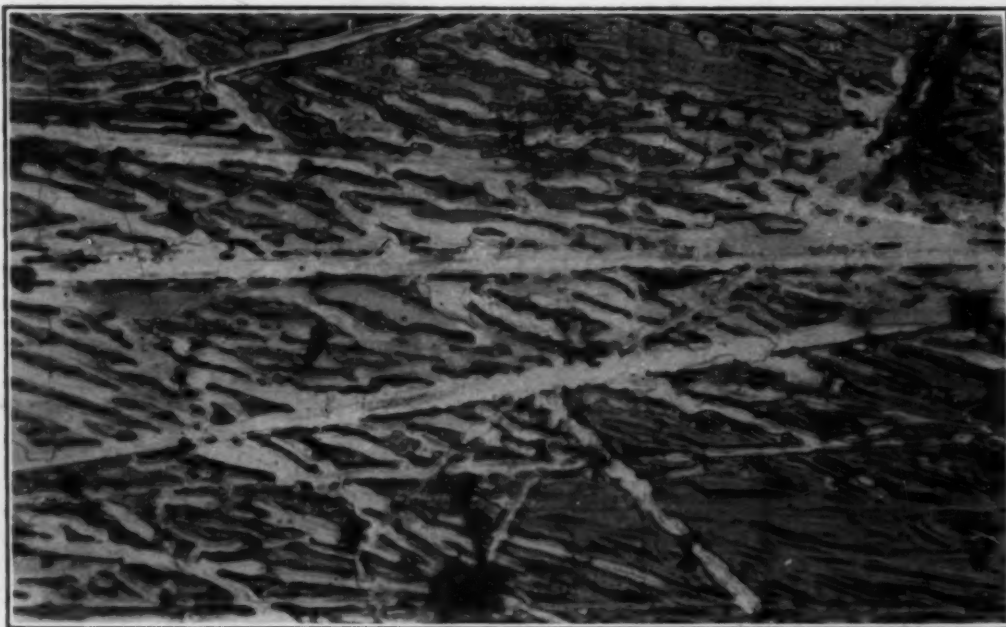


Fig. 4—Silver Filling a Network of Grooves on a Ground Iron Surface (120-Grit Belt, Roughness 23×10^{-6} Inches, $\times 100$).

roughness 65×10^{-6} inches). The sharp boundary between the full and the empty part of the groove is clearly seen. A similar view is shown in Fig. 4, where a meshwork of grooves is covered with silver ($\times 100$, No. 120-grit belt, roughness 23×10^{-6} inches).

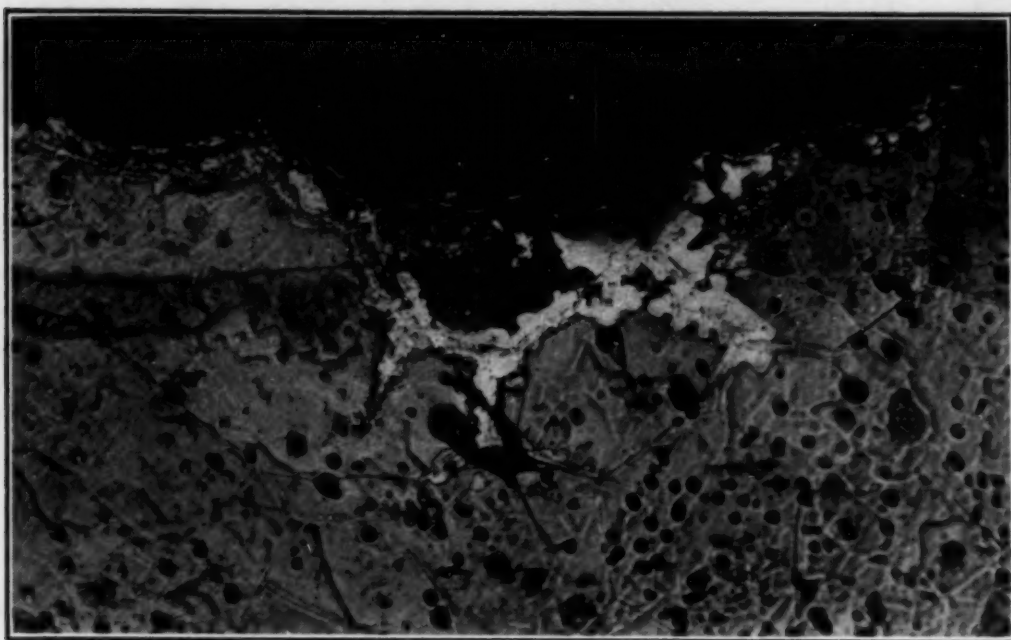


Fig. 5—Silver Spreading Along Grain Boundaries on an Iron Surface (3/0 Emery Paper, Roughness 1.5×10^{-6} Inches, $\times 100$). Solid Mass of Silver Appears Dark Due to an Illumination Effect.

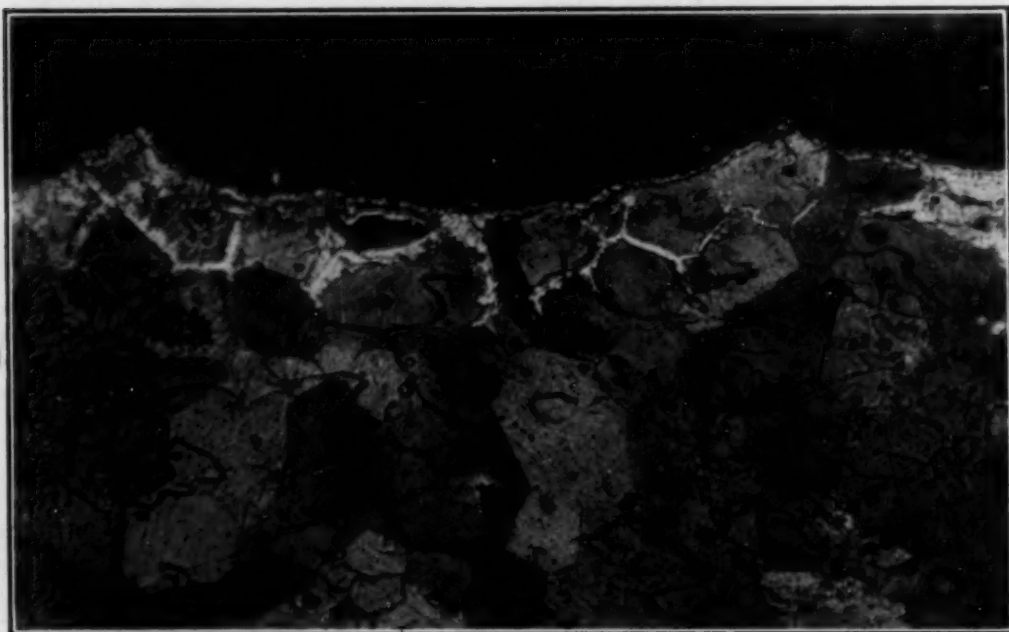


Fig. 6—Silver Spreading Along Grain Boundaries on a Polished and Etched Iron Surface (10 per cent nital, roughness $5.5-6 \times 10^{-6}$ Inches, $\times 100$). Solid Silver Appears Dark Just as in Fig. 5.

Fig. 5 shows a surface finished on 3/0 emery paper (roughness 1.5×10^{-6} in.) on which during heating the heat etching⁴ effect made

⁴O. O. Miller and M. J. Day, TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 541.

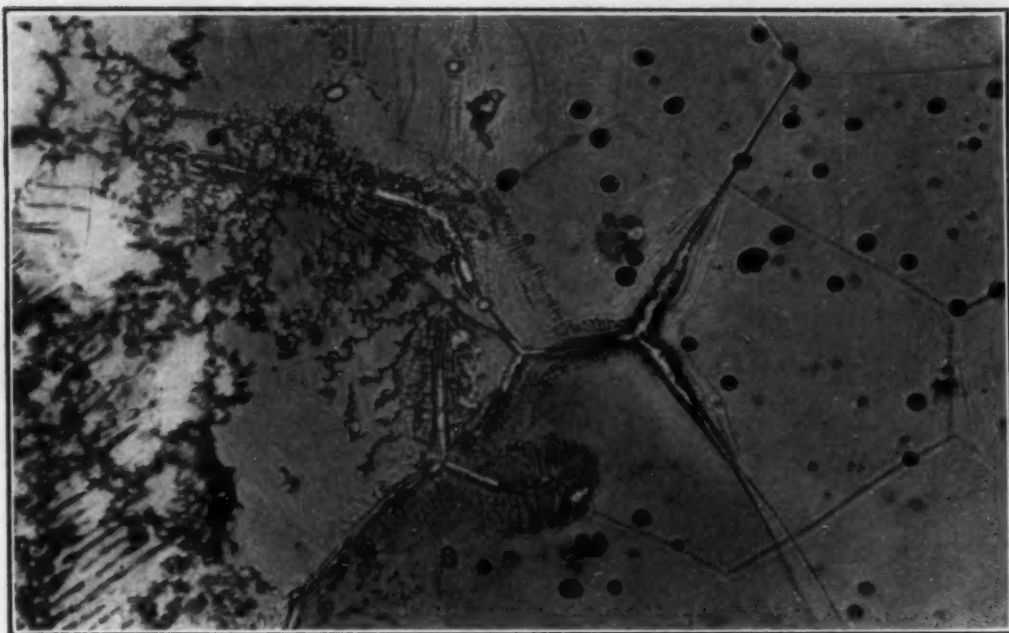


Fig. 7—Silver Spreading on a Polished Surface Along Grain Boundaries Before and After New Grains Were Formed (High Polish, Roughness 0, $\times 100$).

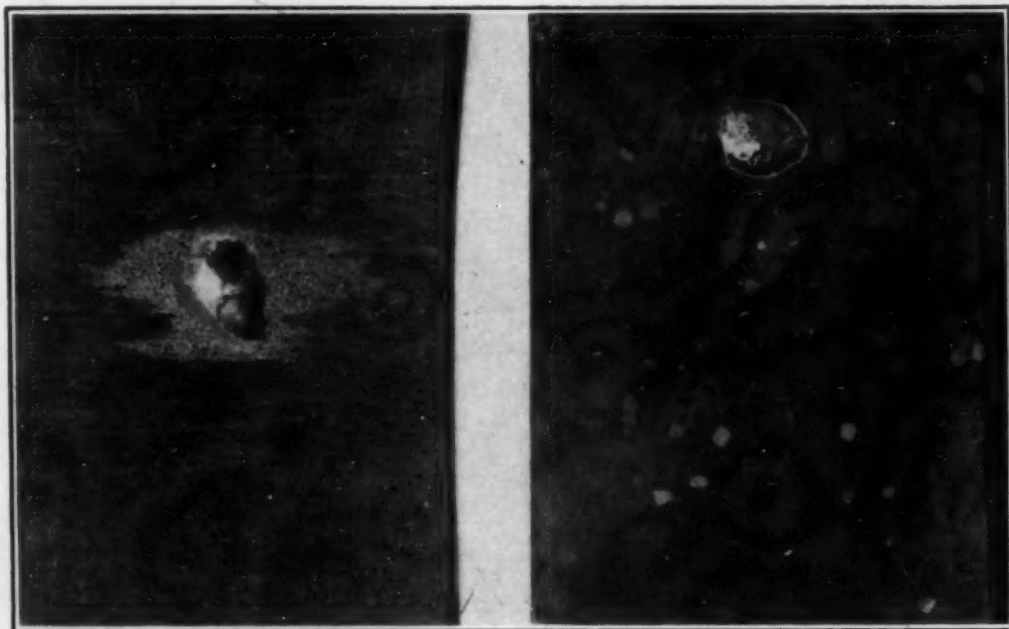


Fig. 8—Silver Spreading on a Roughly Polished (Left) Iron Surface as Compared With a Well Polished (Right) Surface (actual size).

the grain boundaries well visible. These boundaries are narrow grooves along which silver moves forward more rapidly than on the surface of the grains. The difference of color between the big, dark mass of silver on top of the picture and the white silver along grain

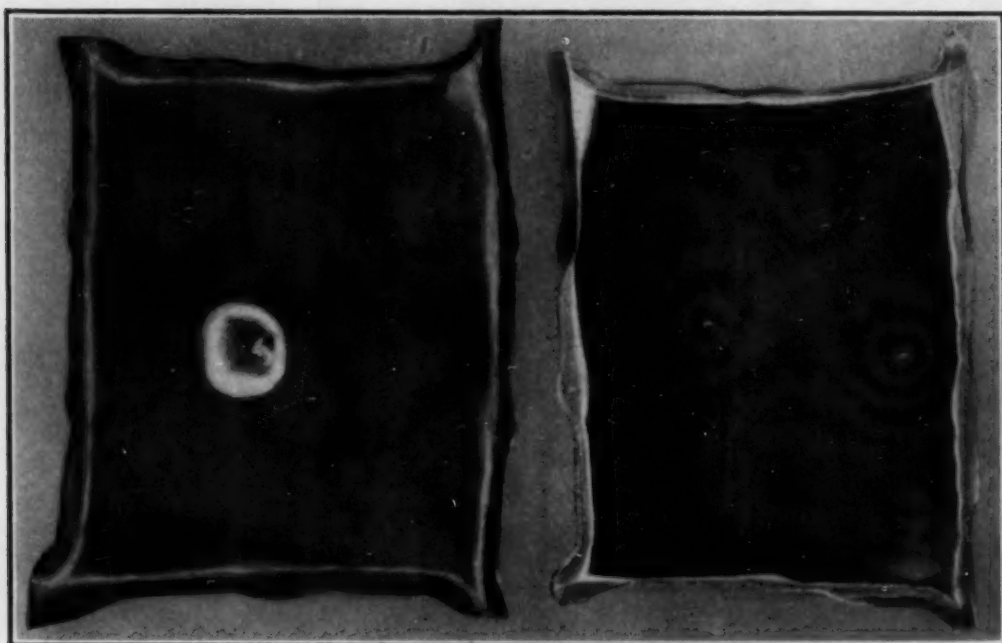


Fig. 9—Silver Spreads Very Little on a Surface Polished with Emery Paper (Left) but It Covers the Whole Iron Boat Which Was Polished and Etched with 50 Per Cent HNO_3 (Right) (actual size). The Silver Covered Boat Appears Dark Due to an Illumination Effect.

boundaries is due to an illumination effect. Fig. 6 shows a similar effect on a surface etched with 10 per cent nital. In Fig. 7 are visible both the old and the new grain boundaries formed by grain growth during heating. Here silver started spreading along the old boundaries and then, after the grain structure had changed, it followed the new boundaries. The fine pattern is due to the presence of some surface oxide which formed during cooling. Fig. 8 shows a typical pattern obtained on a roughly polished surface compared with a well polished surface. The preferential spreading along the grooves is easily visible. Fig. 9 shows the difference in behavior between a sample etched with 50 per cent nitric acid and an unetched surface (both thoroughly cleaned). The silver spread over an area about one half inch in diameter on the unetched sample but spread out into a uniform thin layer on the etched surface. The silver in this case actually flowed up the walls of the iron boat, down the outside, and completely across the bottom, resulting in a sample with a uniform silver plate.

The purpose of these simple considerations and experiments is to establish a rough correlation of the observed facts. It is clear that in a more quantitative analysis of the phenomena involved, other additional factors should be taken into account.

CONCLUSIONS

1. Some of the phenomena observed in brazing and coating can be correlated on the basis of a simple theory which takes into account the roughness of the surface and the surface energies.
2. Cleanliness of a metallic surface is in itself not sufficient to insure spreading of a liquid metal over the surface of the solid.
3. The presence of capillary grooves, machined or etched, and of grain boundaries on metallic surfaces greatly facilitates the spreading of a liquid metal. This depends upon the etching reagent as well as upon the orientation of the grains.

DISCUSSION

Written Discussion: By D. L. Martin, Research Laboratory, General Electric Co., Schenectady, N. Y.

The authors are to be complimented for a very interesting and instructive paper. They have demonstrated clearly the importance of capillarity in any process involving the spreading of a molten metal, as in brazing or dip-coating.

I would like to cite two other instances where capillary action is operating:

1. When a copper-tungsten powder compact is heated to 1200 degrees Cent. (2190 degrees Fahr.), globules of molten copper form on the surface as in Fig. A. Upon heating to a higher temperature or holding a longer period of time at 1200 degrees Cent. (2190 degrees Fahr.) the liquid is drawn back into the sample to leave the surface smooth as in Fig. B. This strange phenomenon was observed many years ago but was not explained until the authors' work on capillarity was made known. The molten copper is drawn back into the porous compact because of capillary action.

2. Since the pores or channels between powders in a pressed compact act as capillary tubes it should be possible to make an alloy by capillary penetration. For example, if a pressed cylinder of iron powder is placed on a copper sheet, as is illustrated in Fig. C, and the whole assembly heated to 1200 degrees Cent. (2190 degrees Fahr.), we might expect the copper to penetrate into the iron mass. The photomicrograph in Fig. D shows that is exactly what occurred when the experiment was performed. The molten copper was drawn up into the pores of the iron compact to form a very dense alloy.

Oral Discussion

C. J. BIER:⁵ The previous comment on copper-iron briquettes has brought up the question of the influence of capillarity on powder metallurgy techniques.

That sintering operation which relies on one metal powder which becomes molten to bond a second metal powder which remains solid is concerned with this subject. However, a complex three-dimensional capillarity is observed, since the green briquettes are porous. Very little is known about the mechanism of this reaction.

⁵Director of research, H. L. Crowley and Co., Inc., West Orange, N. J.

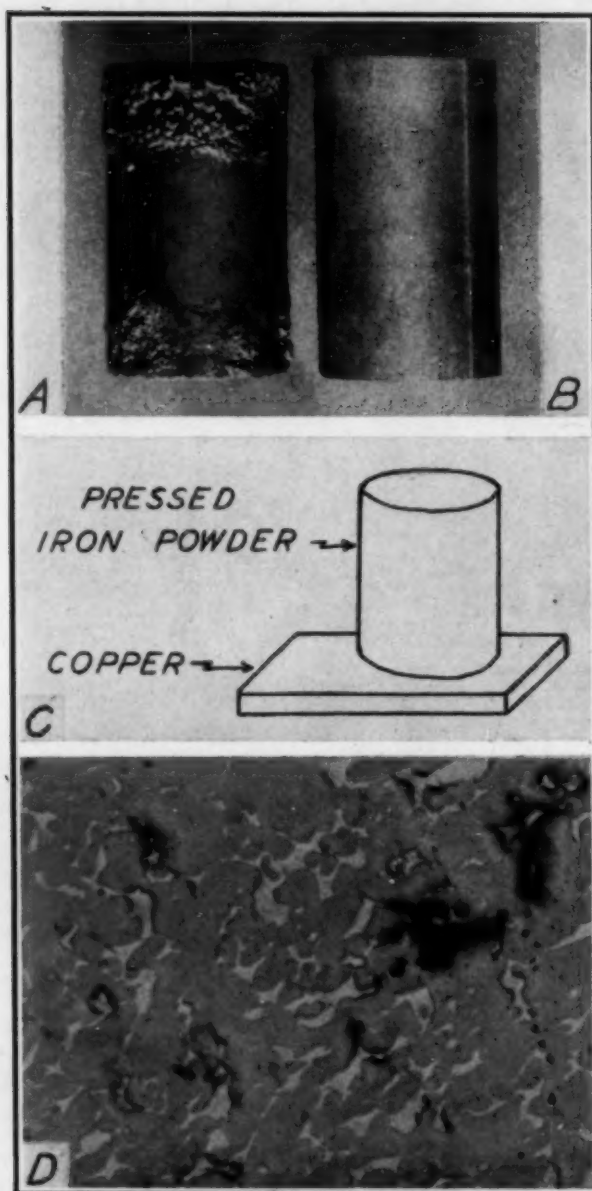


Fig. A—Appearance of Copper-Tungsten Powder Compact. Heated for a Short Period of Time to 1200 Degrees Cent. $\times 1$. Note the Excess Copper on the Surface. Fig B—Same as Fig. A but Heated a Longer Period of Time to Allow the Copper to be Drawn Back into the Sample.

Fig. C—Copper-Iron Alloy Made by Capillary Penetration of Copper into Pressed Iron Powder. Sketch of Assembly. Fig. D—Photomicrograph Taken Near the Top of the Sample Showing that the Copper (Light Areas) Has Flowed into the Pores Between the Sintered Iron Particles. $\times 250$.

The work reported by the authors in their enlightening paper has been a good start on the general subject of the capillarity of metal surfaces. It is hoped that their further studies will be concerned with some of the problems of capillarity encountered in powder metallurgy.

Authors' Reply

Dr. Martin has given us some very interesting examples of metallurgical experiments in which capillarity plays an important role. A closer investigation of the "sweating out of copper" and the subsequent "clean-up effect" shows that the sweating occurs just above the melting temperature of copper and it occurs all over the surface of the pressed tungsten-copper sample. The reason for this sweating is probably thermal expansion of copper which at the melting point has low viscosity and cannot penetrate into the thin pores. At higher temperature, viscosity of copper decreases rapidly and the capillary forces suck it back into the porous block. In Fig. A copper has already disappeared in the central part of the block either because of a slightly higher temperature or because of the usually lower density (larger pores) in that part of pressed blocks.

Mr. Bier points out the very interesting and complex problem of capillarity in powder metallurgy. In order to make a quantitative study of the role of capillary forces in sintering, one needs first a proper estimate of the size and distribution of porosities in pressed powders. Then a series of sintering experiments under controlled conditions can give the right answer. Work along these lines is partly in progress.

A COMPARISON OF MICROHARDNESS INDENTATION TESTS

BY DOUGLAS R. TATE

Abstract

The Tukon and Eberbach testers have extended the range of hardness tests to small areas, thin surface layers and brittle materials. The indentation numbers obtained with these machines are not, however, independent of load, a fact which makes advisable an examination of the reasons for this lack of uniformity. Comparison data for the two testers throughout their common range of loads are presented and, in addition, theoretical reason and experimental evidence is advanced to show that Knoop indentation numbers are not based on unrecovered indentation areas. Recommendations are made for the specification of indentation numbers.

INTRODUCTION

NEW fields of metallurgical research have been opened by the development of the Tukon tester and the Eberbach tester, each having different methods of applying load but covering a comparable range of loads. Both of these testers have found uses in production control; and already specifications have been written containing requirements for the Knoop number.

It has been noted by several observers¹ that for a number of materials the Knoop number is consistently slightly higher at low indenting loads. This has been ascribed to work hardening of the surface during preparation of the specimen. The fact that no effect of similar magnitude has been observed in indentations made with the Vickers indenter under low loads raises the question of whether the increase in Knoop number is due to properties of the materials or to some characteristic of the test itself.

If the effect is due to some characteristic of the test, this factor

¹Constance B. Brodie, "The Microhardness Tester as a Metallurgical Tool," *TRANSACTIONS, American Society for Metals*, Vol. 33, 1944, p. 126.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, Douglas R. Tate, is associated with the Engineering Mechanics Section, National Bureau of Standards, Washington, D. C. Manuscript received June 2, 1944.

should be taken into account in the interpretation of results which involve the comparison of indentation numbers calculated from indentations made under different indenting loads. The purpose of this paper is to examine results obtained with the two microhardness testers mentioned previously, and to compare their performance at low loads.

The results are based upon tests made at the National Bureau of Standards, in which both Knoop and Vickers indenters were used under controlled conditions.

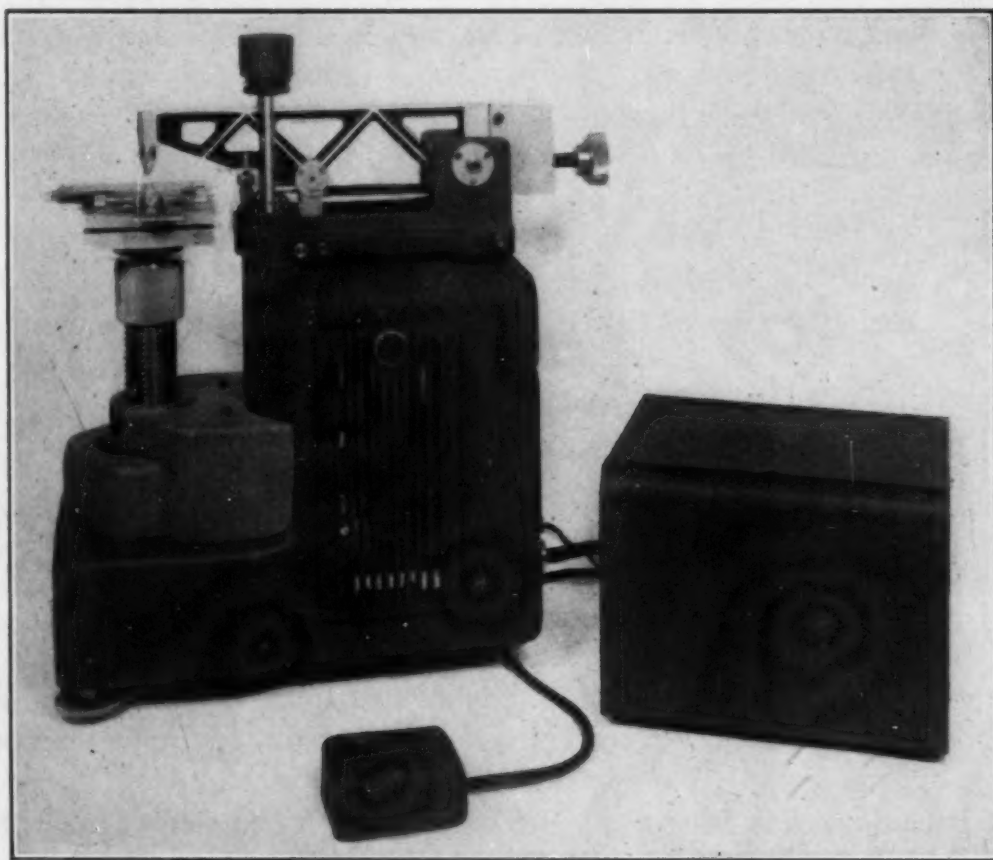


Fig. 1—The Tukon Tester Showing the Control Box.

THE TUKON TESTER

The Tukon tester, Fig. 1, consists of a counterbalanced beam supported by an insulated contact and a fulcrum knife-edge. An indenter is attached at one end of the beam and a weight hanger is suspended from a knife-edge midway between the indenter and fulcrum knife-edge.

The specimen is placed on a table carried by an elevating screw which is actuated by a reversible motor and worm gear.

In operation the specimen is placed on the table and a button momentarily depressed. The elevating screw motor starts driving the elevating screw upward and continues until the specimen contacts the indenter and lifts the loaded beam free of the insulated contact support. After a timed interval, a lifting fork automatically lifts the beam and indenter free of the specimen while the elevating screw motor lowers the specimen. The beam is then returned to its insulated support and the cycle is complete.

A set of weights is included providing a range of indenting loads from 3600 to 100 grams. If desired, the range may be extended to 25 grams by means of a special hanger. This tester is now produced with a solenoid device to eliminate errors due to the inertia of the beam and weights as well as a spring device to prevent play of the elevating screw in its bearing.

Although the Tukon tester is supplied with the Knoop indenter², it can easily be adapted to the 136-degree Vickers diamond pyramid indenter.

THE EBERBACH TESTER

The Eberbach tester, Fig. 2, consists of a spring loaded Vickers indenter moving axially in a bearing which may be attached to the barrel of a microscope in place of the standard objective lens mount. In making a test the indenter is lowered onto a specimen on the microscope stage by the fine adjustment. An electronic device indicates when the full indenting load is applied to the specimen.

An Eberbach tester used for the tests reported in this paper was equipped with six interchangeable helical springs giving a range of indenting loads from about 835 to 26 grams when calibrated for a vertical type microscope. If calibrated for a microscope of the type in which the specimen is viewed from below, this tester would have had a range from about 817 grams to 7.5 grams. The difference is due principally to the weight of the indenter spindle and springs.

CALIBRATION

Three Tukon testers in use at the National Bureau of Stand-

²F. Knoop, C. G. Peters and W. B. Emerson, "A Sensitive Pyramidal-Diamond Tool for Indentation Measurements," *Journal of Research*, National Bureau of Standards, Vol. 23, July 1939, p. 39.

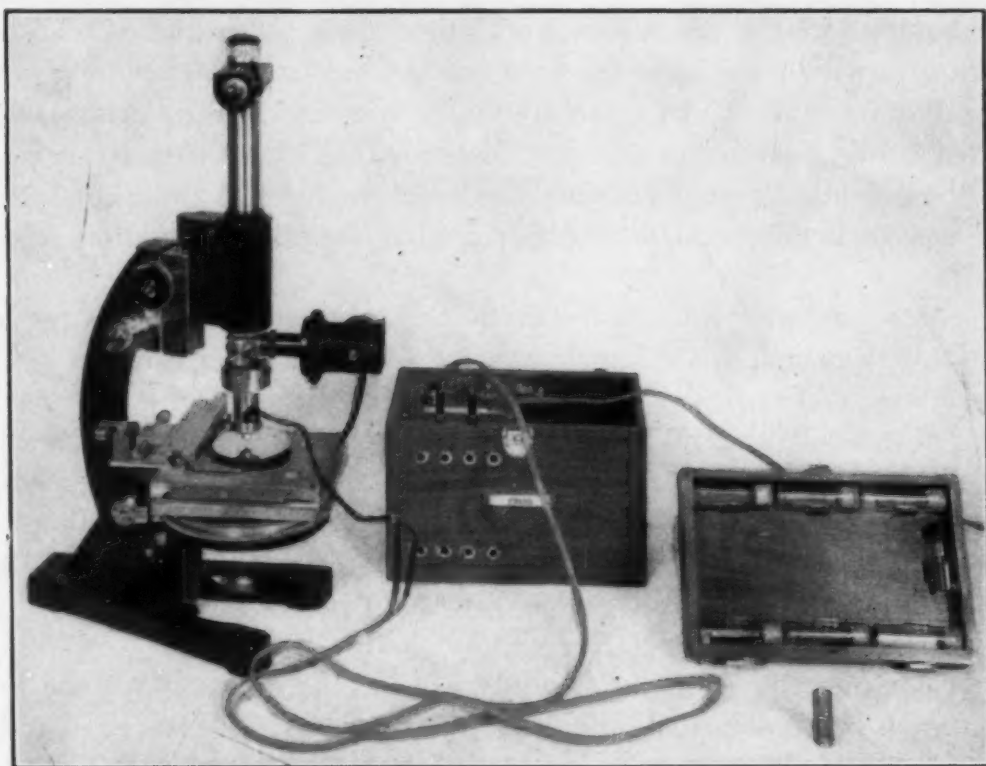


Fig. 2—The Eberbach Tester Mounted on a Vertical Type Microscope Showing the Contact Indicator Box and One of the Loading Springs.

ards have been calibrated using an equal arm balance and an electronic contact indicator. The errors in the indenting loads were less than 0.3 per cent for all three machines. The errors due to the balance and weights did not exceed 0.01 per cent. The indenter complied with the National Bureau of Standards tentative specifications for Knoop indenters.

The Eberbach tester was calibrated using similar equipment. This tester, according to the manufacturer's instructions, should be calibrated each time one of the load applying springs is removed from the device. If this precaution is observed, the indenting load may be known to within about 1 per cent unless the device is jarred or the spindle and guide allowed to become dirty. No tests were made to determine how this indenting load would change with time. Probably the device should be calibrated before using it each day.

If the manufacturer's instructions regarding calibration each time a spring is removed from the tester are disregarded, errors as great as 4 to 5 per cent in the indenting load may be expected.

Provided the machines are in proper working order and are calibrated, the principal source of uncertainty in the determination

of Knoop and Vickers numbers with the Tukon tester and of Vickers numbers with the Eberbach tester lies in the measurement of the indentation lengths. An error of 1 per cent in measurement of an indentation will lead to an error of twice that amount or 2 per cent in the indentation number, since the length of the indentation appears as a second power factor in the formulas for the indentation numbers.

A micrometer microscope used for such work should always be calibrated against some known standard. Even when this has been done, inequalities in the skill and eyesight of different observers may lead to considerable differences in results reported for the same indentation. The microscope should be used at the highest magnification practicable, in order to resolve the ends of the indentation to the fullest extent. The micrometer microscope employed in the measurement of the results quoted in this paper was checked with a calibrated stage micrometer.

The uncertainty in the measurement of the diagonals of the indentation is the principal objection to the use of the Vickers indenter at low loads. The Vickers indentation is so small that it is difficult to obtain reproducible results even with an excellent surface finish. The Knoop indentation is about three times as long as a Vickers indentation made under the same load and in the same material.

EFFECT OF LOAD ON INDENTATION HARDNESS

Three specimens were prepared by the Optical Shop of the National Bureau of Standards using low speed wet hand lapping methods and were polished to optical finish on low speed wet cloth and pitch laps. The specimens included a Rockwell test block, a block of stainless steel of the cutlery type heat treated to about Rockwell C-48 and a block of medium flint optical glass.

The indenting loads ranged from 1000 to 25 grams except for the Vickers tests on glass for which they ranged from 100 to 25 grams. The results are shown graphically in Fig. 3. Each point represents the average of ten indentations made in a calibrated Tukon tester.

The results clearly show an increase in Knoop number with low loads, particularly in hard materials, and a similar but less pronounced effect with the Vickers indenter. The curves for the Knoop number for these three rather different materials possess such a

noticeable similarity that one might conclude that the effect is due not so much to the physical characteristics of the materials as to the characteristics of the test itself.

Knoop, Vickers, Brinell and other less common indentation numbers are defined as the average stress over an area associated with the indentation. The deformation produced by a loaded in-

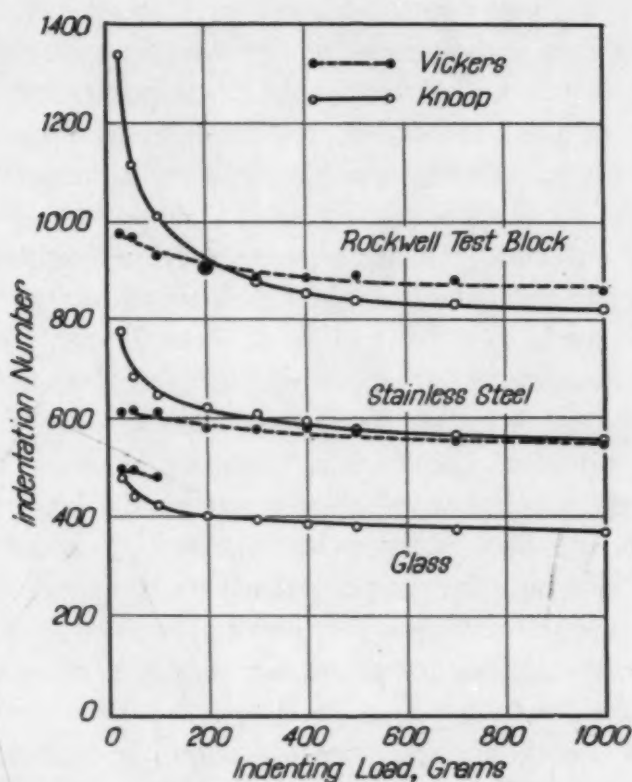


Fig. 3—The Relation of Indentation Number to Indenting Load.

denter is in part elastic and in part plastic. Upon removal of the indenter an appreciable part of the elastic deformation disappears. The amount of elastic recovery is dependent on factors such as the susceptibility of the materials to cold working, shape of the indenter and indenting load.

In the case of the Knoop indenter, Knoop, Peters and Emerson² assumed that since the major portion of the elastic recovery of a Knoop indentation takes place along the shorter diagonal, the elastic recovery of the long diagonal could be considered negligible. The indentation number determined by a measurement of the long diagonal would then be an unrecovered indentation number. That is, an

indentation number calculated on the basis of the area of contact of indenter and specimen when under load. Further consideration, however, indicates that the recovery of the long diagonal may not be a negligible factor.

The material at the end of the long diagonal of a Knoop indentation has been strained sufficiently to produce a permanent deformation. If this were not so, there would be no indentation to see in the microscope at that point. However, since the amount of deformation decreases with increasing distance from the center of the indentation, it is not an unreasonable assumption that immediately beyond the tip of the indentation lies material which was deformed elastically by the loaded indenter and recovered when the indenter was removed.

If this is so, and if a Knoop indentation could be observed under load, the long diagonal would be observed to shorten upon the removal of the load.

Since the geometrical relationship between the surfaces of the indenter and the specimen at the ends of the diagonals of the indentation is independent of the load, it might be expected that for any given specimen the amount of shortening would be, to a first approximation, independent of the indenting load. A constant recovery such as this would be a larger percentage of the total length of the long diagonal in small indentations than in the case of larger indentations and would cause an apparent increase in hardness in indentations made at low loads.

This same theory applies also to Vickers indentations, but the change in length would be only about one-fifth as great because of the larger angle between the edge of the Vickers indenter and the surface of the specimen.

In order to determine approximately the recovery of a Knoop indentation upon removal of the indenting load, the following experiment was performed.

A glass specimen was mounted on the stage of a microscope so arranged that a moderately high powered objective lens giving a total magnification of about $\times 400$ could be focussed on the bottom surface of the specimen, i. e., looking at that surface through the specimen. A Knoop indenter was brought into contact with this surface from below by means of a lever operated by a suitable slow motion device, so that the indentation could be continuously viewed and measured while the loaded indenter was in contact with the in-

dentation, the magnitude of the load being determined by the lever ratio and the position of a dead weight.

Three series of ten indentations each were measured under load and immediately after removal of the load. The glass specimen was not the same glass as in the specimen of Fig. 3. The results of the experiment are given in the table.

Series	Length of Long Diagonal		Difference Microns
	Under Load Microns	After Removal of Load Microns	
1	90.8	85.4	5.4
2	92.4	87.4	5.0
3	68.7	64.2	4.5

The results clearly indicate a recovery of about five microns. An interesting feature of this experiment was the appearance of a series of five or six interference bands extending out beyond the tip of each long diagonal of the Knoop indentation while the loaded indenter and specimen were in contact. The spacing of these bands, which were evidently formed between the edge of the indenter and the flat surface of the specimen, indicated that the specimen was quite flat along the extended line of the long diagonal.

Taking five microns, the average of the three entries in the last column of the table, as the best available estimate of the recovery of a Knoop indentation diagonal, it is interesting to note the effect of applying this as an adjustment to the data for the Knoop curves in Fig. 3. The results of this adjustment are shown in Fig. 4. The adjusted curves are good approximations of constant values to about 3 per cent.

Since the amount of elastic recovery of the long diagonal is small, its effect should be negligible in indentations made under very heavy loads. The Knoop numbers of the Rockwell test block and the stainless steel block were determined under loads of 100 kilograms in a calibrated Vickers machine. Under this load the Knoop number was 770 for the Rockwell test block and 512 for the stainless steel block. These values agree with the average values of the adjusted data, Fig. 4, to about 3 per cent for the stainless steel and better than 1 per cent for the Rockwell block. This indicates that if this adjustment is made the Knoop number for these specimens remains constant to within about 3 per cent for loads varying from 25 to 100 kilograms, a ratio of 4000 to 1.

This would seem to be fairly definite evidence that elastic recovery of the long diagonal of a Knoop indentation does take place

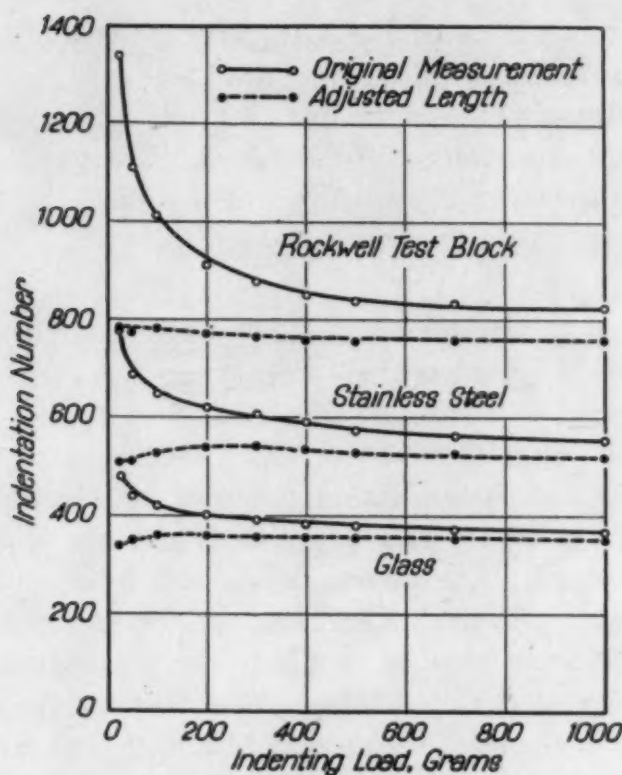


Fig. 4—Effect of Adding 5 Microns to Each Knoop Indentation Length.

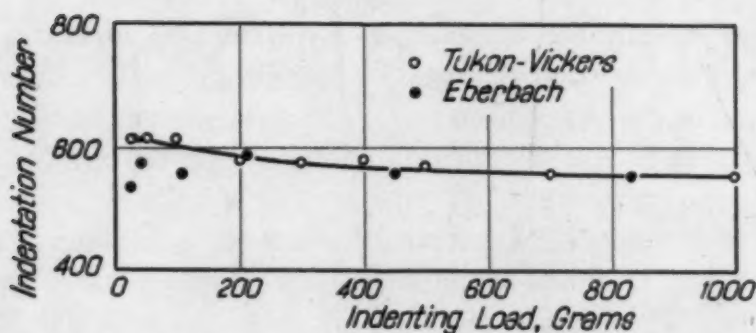


Fig. 5—Comparison of the Eberbach Tester with the Tukon Tester Equipped with a Vickers Indenter on Stainless Steel.

upon removal of load in metals as well as in glass. If this is so, recognition of the fact should be made in the interpretation of experiments on hardness gradients, where indentation numbers obtained under different loads are compared with one another. It is, moreover, evident that an indentation number can have little meaning unless the indenting load in addition to the indenter is specified.

The desirability of including a statement of the load with the indentation number has already been pointed out by Mr. Erle J.

Hubbard in a discussion of a paper by Mrs. Constance B. Brodie.¹ It is not proposed here that data obtained with Knoop indenters be corrected for the elastic recovery effect. It appears preferable to compute indentation numbers in the usual manner and to state the value of the indenting load.

COMPARISON OF TUKON AND EBERBACH TESTERS

Ten indentation tests were made with each of the six load springs in the Eberbach tester on the stainless steel block for comparison with the Vickers indenter in the Tukon tester. The results of the comparison are shown in Fig. 5. Since the errors observed during calibration for load were not sufficient to explain the poor agreement of the results below 200 grams, it is believed that they are due to experimental errors of measurement. The results obtained with both testers were in good agreement at higher loads.

SUMMARY

Both the Tukon and Eberbach testers are satisfactory devices for applying indenting loads of 1000 grams and less. The Tukon tester can be used with both Vickers and Knoop indenters when a suitable adapter is provided, and the two machines give results in good agreement for indenting loads of 200 grams and greater.

There is reason to believe that the Knoop number of a specimen increases with decreasing loads due to elastic recovery in the long diagonal. This would occur in a perfectly homogeneous specimen, although some surface hardening is probably always present. The results of an indentation test made at low loads must be regarded from the operational viewpoint. They are functions dependent on the indenting load and have little meaning without specification of the indenting load.

DISCUSSION

Written Discussion: Erle J. Hubbard, assistant to chief metallurgist, Koppers Co., American Hammered Piston Ring Division, Baltimore, Md.

The information and data presented in Mr. Tate's paper collaborate the work being done in our Research Laboratory on the correlation and relationship between the Knoop hardness and Rockwell hardness numbers. A considerable amount of work has been done which reveals that the lower loads on the Knoop indenter give considerably higher Knoop numbers. A number of explanations have been offered as to the cause of this.

Our investigations to date, although not complete, indicate that materials which normally show a higher Knoop hardness number appear to show a greater percentage of increase in hardness at the lower indenter loads than do softer materials. For example, a homogeneous material recently investigated and having a Rockwell 15-N hardness of 90.5 to 91.5 showed a drop in hardness from 912 Knoop hardness numbers at 100-gram load to 780 Knoop hardness at 1000-gram load. Whereas, a homogeneous material showing a Rockwell "C" hardness of 23.5 to 25.5 only showed a drop from a Knoop hardness of from 317 at 100-gram load to 294 at 1000-gram load. In the first case, the drop represented approximately a 14 per cent difference in hardness; in the second case, approximately 7 per cent drop in hardness. Nevertheless, the corrections applied to these hardness numbers as determined by Mr. Tate gave fairly consistent results as shown in the following table:

Material No. 1		
Rockwell 15-N Hardness 90.5-91.5		
	Length of Indentation mm	Knoop Hardness
100-gram load before correction.....	0.0396	912
1000-gram load before correction.....	0.1351	780
100-gram load after correction.....	0.0446	715
1000-gram load after correction.....	0.1401	725

Material No. 2		
Rockwell "C" Hardness 23.5-25.5		
	Length of Indentation mm	Knoop Hardness
100-gram load before correction.....	0.0670	317
1000-gram load before correction.....	0.2200	294
100-gram load after correction.....	0.0720	274
1000-gram load after correction.....	0.2250	281

From the information presented by Mr. Tate, it appears evident that the elastic recovery of the long diagonal is not a negligible factor. Therefore, it appears to me that the Knoop hardness number tables need a revision or new tables be formed which would apply to the Knoop hardness numbers when loads less than 1000 grams are used on the Knoop indenter.

Mr. Tate mentions that he does not propose that this be done and it is true that it is probably not necessary if the value of the indenting load be given. However, we have found that occasionally there is some question of Knoop hardness numbers when trying to convert these hardness numbers in terms of Rockwell "C" hardness or other hardness numbers more familiar to the industry.

We have our own conversion tables made in such a manner that no matter what load is used on the Knoop indenter, we can determine what the corresponding Rockwell "C" hardness is within allowable tolerances. The setting up of these tables is quite an undertaking and offers many chances for experimental errors.

We have had little experience with the Eberbach tester but it has its applications the same as does the Tukon tester. We have, however, attempted to use the 136-degree diamond with attachments for the Rockwell superficial tester and find that the impressions are difficult to read and, therefore, experimental errors are introduced.

Written Discussion: By A. L. Rustay, assistant chief metallurgist, Wyman-Gordon Co., Worcester, Mass.

The Tukon hardness tester in use in our laboratory has been calibrated against Rockwell test blocks of various hardnesses. The results have shown that lighter load consistently gives higher hardness numbers. Fig. A shows that our data are in agreement with those obtained by Mr. Tate, as far as the general trend is concerned, when a correcting factor of 5 microns is added to the length of each indentation.

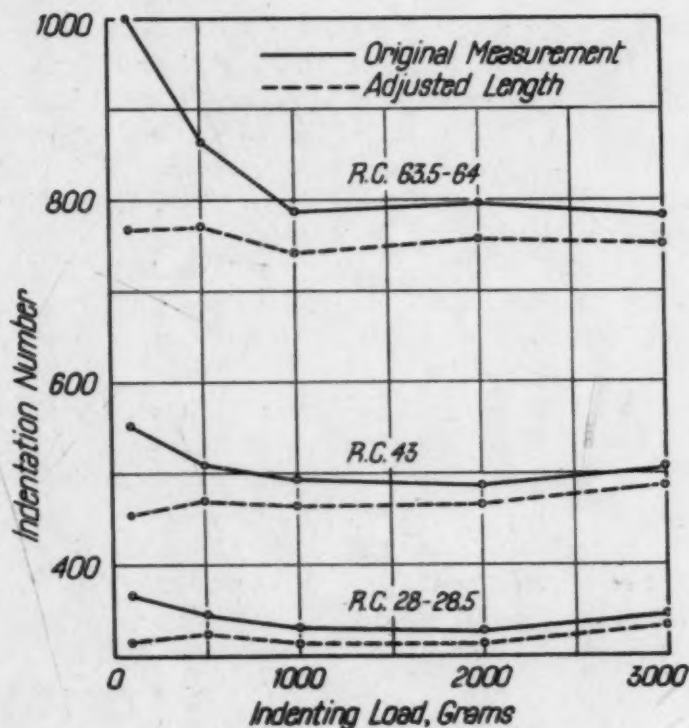


Fig. A

The hardnesses listed in the chart are the average of a number of readings, made by one person, of indentations made with a machine not equipped with the inertia correcting solenoid.

Written Discussion: By L. P. Rice, metallurgist, Bendix Products Division, Bendix Aviation Corp., South Bend, Ind.

Mr. Tate has presented some interesting and timely data on microhardness testing. The variations in hardness numbers encountered with loads under 1 kg. are apparently explained and the usefulness of the Knoop indenter with small loads is greatly enhanced.

This paper again makes us aware of the many peculiarities and factors involved in indentation hardness testing. Elastic recovery cannot be ignored

even with the Knoop indenter, as was previously believed. In Fig. 4 of the author's paper, the shape of the corrected curves indicates that perhaps elastic recovery is not the only factor involved. There probably is some uncertainty in determination of the exact ends of the long diagonal of the Knoop indentation in hard materials using a low load. Also, would not the elastic recovery change with the modulus of elasticity of the material under test?

It is interesting to note that the microhardness numbers as obtained by the use of the two types of indenters are fairly comparable. This is true even though the basis for the mathematical determination of the hardness numbers is not on a strictly comparable basis. The Vickers hardness number is based on the area of contact between indenter and specimen using the measurement of the recovered indentation, whereas the Knoop hardness number is based on the "unrecovered" projected area of the indentation.

If we base the Knoop hardness number on area of contact and compare this number with the Vickers numbers for the same material, we find a greater spread between the hardness numbers. This spread or difference in hardness number will increase as the hardness of the material tested increases.

With the correction for recovery of the Knoop indentation, the difference in hardness numbers, regardless of how they are calculated, is made fairly uniform for the various loads used. The reason for this difference is probably due to the geometry of the two indenters.

The Knoop and Eberbach hardness tests will, no doubt, find increasing use and will add to our interest in the theoretical aspects of hardness testing.

Written Discussion: By Charles A. Nagler, instructor of metallography, University of Minnesota, Minneapolis, Minn.

The method used to study the recovery of a Knoop indentation upon removal of the indenting load was clever and interesting. According to the data gathered from the measurements, the recovery of the length of the long diagonal was approximately 5 microns. What loads were used in the three series of ten indentations made to note recovery? It would be interesting to know what effect or changes would be made on the curves for the Knoop tests shown in Fig. 3 if the correction for recovery were applied for the range of loads studied. In gathering the data for the curves in Fig. 4, was the assumption made that this recovery of 5 microns would apply to all loads? In comparing Fig. 4, indentation number on Knoop for the Rockwell bar, with the newly calculated indentation number, with that of Fig. 3, for the Vickers indentation number, the indentation numbers are not in good agreement.

In working with chrome plated surfaces the generally used test has been to determine if the coating is file hard. In our laboratory attempts were made to establish Vickers indentation numbers, determined by the Eberbach tester, for this chrome plate. As was anticipated, the higher indentation numbers were characteristic of a chrome plate that was file hard, whereas the lower numbers were characteristic of chrome plates which were not file hard.

In making the determination of the length of the diagonal it was necessary to magnify the impression 1000 times. In the calculation for the Vickers hardness number the following formula is used:

$$H = 1.8544 \frac{L}{d^3}$$

where H = Vickers number, L is the load in kilograms, and d is length of the diagonal in millimeters. An error in the measurement of the length of the diagonal is magnified in that in calculating the Vickers number the length of the diagonal is squared. An error in measurement of the length of the diagonal is magnified many fold in the actual hardness number calculated therefrom.

In determination of hardness of chrome plate the length of the diagonal impressions varies from 5.5 to 10 millimeters at 1000 magnification which would when calculated show a Vickers hardness number of 1405 on the high side and 421 on the low side. In the hardness determinations the load in kilograms on the diamond was 0.02273.

What have been your experiences, if any, in determining the hardness of chrome plate using either the Knoop hardness tester or the Eberbach hardness tester?

Written Discussion: By S. R. Williams, professor of physics, Fayerweather Laboratory of Physics, Amherst College, Amherst, Mass.

The investigation which Mr. Tate has made is an interesting one, because it is dealing with microhardness testers, and the writer of this discussion believes that microhardness testers offer some special advantages over others as research tools.

There is one question, however, to be raised about all inter-comparisons of hardness testers and the obtaining of hardness conversion relationships: What effect does the anisotropy of the material being tested have upon the values used in the comparison work?

In a paper which should have been ready, and is not, it has been shown that in making Brinell tests on cold-rolled nickel, brass and steel a variation of as much as 3 per cent exists between the Brinell hardness number when measured along a diameter parallel to the direction of rolling as compared with that at right angles to the same.

The author hopes that when the war is over he will have an opportunity to use the Knoop instrument on cold-rolled materials. Will the Knoop instrument minimize or exaggerate the effect of the anisotropy of cold-rolled materials?

Apparently little attention has been paid to the effect of anisotropy in the many comparisons on hardness tests by different testers to be found in the large number of studies recently appearing on the comparison of hardness numbers. The older writers had a lot to say about the anisotropy of crystals in measuring their hardness. It is a subject deserving further study.

Written Discussion: By Aaron M. White, associate metallurgist, War Department, Watertown Arsenal, Watertown, Mass.

This paper is another contribution to the information concerning Knoop hardness measurements and serves to explain the anomaly whereby higher hardness numbers are obtained for lower indentation loads. While the author's approach to the problem of determining the amount of recovery is straightforward it appears that he may have made an error in assuming that the amount of elastic recovery that would take place in steel is the same as would occur in glass. Would not the amount of recovery depend upon the elastic modulus of the material?

Written Discussion: By Constance B. Brodie and R. Smoluchowski, Research Laboratory, General Electric Co., Schenectady, N. Y.

The author is to be congratulated for his interesting contribution to the better understanding of the determination of hardness with these testers.

We are particularly interested in Mr. Tate's results which show an increase in hardness when using decreasing loads. We have obtained the same relation between load and hardness numbers on a similar Rockwell test block but have found the relation does not hold for all metals. This is shown in Table A.

Table A
Knoop Hardness Numbers for Various Loads*

Load in Grams	25	50	100	200	500	1000
Steel Rockwell Test Block	1350	1140	1083	970	855	795
0.40 C Steel	810	975	846	740	756	706
0.80 C Steel	570	550	665	600	580	547
Tungsten	495	463	393	340	318	300
Steel Rockwell Test Block	333	344	310	298	305	295
Brass Rockwell Test Block	183	191	188	182	185	164
Copper	58	55	58	52	50	59
Aluminum	19	19	22	18	17	

*The Tukon tester used for the above determinations is equipped with the solenoid device to eliminate errors encountered at the lower loads.

It is evident from the above table that the Rockwell C-68 test block and the tungsten sample show a marked increase in hardness values with decreasing load, whereas the Rockwell C-23 test block and 0.40 carbon steel show less. The difference in values is negligible for the other metals listed. It follows that when interpreting Knoop hardness numbers not only the load but also the type of metal tested should be considered as the deviations at low loads are not the same for all materials. This increase in hardness value when the lighter loads are used cannot be accounted for by work hardening of the surface during polishing, since the hardness numbers of electrolytically polished surfaces are the same as those of a surface covered with a layer of disturbed metal due to poor polishing technique.

In connection with the very interesting results of Mr. Tate's investigations of the recovery of the Knoop indentation in glass, we would like to mention briefly our recent study of recovery of metal during microhardness testing.

The problem is to determine the size of the indentation in metal before and after the diamond is removed. After trying out various methods, the following procedure turned out to give satisfactory results: First the diamond is covered with a thin layer of graphite suspended in water and the indentation obtained in the usual manner in the Tukon microhardness tester. The diamond is removed from the tester and observed under the microscope with micrometer eyepiece. The diamond point appears now clean and shiny with the graphite pushed away during test. The edge of the graphite layer is sharp and the long diagonal of the rhomb can be accurately measured. Subsequently, the indentation itself is measured. The difference between the diagonals measured on the diamond itself and on the metal indicates the recovery of the metal. Important factors in the method here described are the best thickness of the graphite layer and a proper illumination.

The results of our study will be described more extensively in a forthcoming paper. Here it may be pointed out that recovery has been observed for metals as different as copper and tungsten, the magnitude of the recovery is of the order of several microns, similar to Mr. Tate's results, and it seems to be dependent upon the load. Further measurements are in progress.

Written Discussion: By J. M. Berry and G. M. Foley, Battelle Memorial Institute, Columbus, Ohio.

Mr. Tate's measurement of the elastic recovery of a Knoop hardness impression in glass is a very interesting and useful piece of work. It demonstrates clearly the care which should be used when making the theoretical generalizations which led most of us to believe the Knoop impression to be particularly free from elastic recovery in the long direction.

The paper shows that elastic recovery may be expected in any Knoop impression. It is not reasonable, however, to expect that this recovery will be equal in glass and in metals. There are good reasons for believing that the elastic recovery in glass will be greater than in steel on account of the high short-time elastic limit and the low modulus of elasticity of glass.

If Mr. Tate's correction for the elastic recovery of the impression in steel is too large, then there may be evidence in the results of Knoop hardness tests tending to show the existence of a surface layer hardened by cold working during preparation of the metal specimen.

Author's Reply

The author wishes to express his thanks to those who have contributed to this paper by their discussions.

In reply to Messrs. Berry and Foley and also to Mr. Rice and Mr. White, it is quite true that one would expect the amount of elastic recovery to be affected by a difference in the modulus of elasticity and by the time-flow characteristics of the material. In addition, the cold working properties and the elastic range of the material may have considerable influence. For these reasons and also because of the relatively large observational errors encountered in measuring the elastic recovery, the value of five microns can be considered only an approximation. This is one reason why it is recommended in the text that data obtained with Knoop indenters be used without correction for the elastic recovery effect.

In reply to Mr. Nagler, the loads applied to the Knoop indenter in the experiment to determine elastic recovery in the glass block varied from 150 to 250 grams. In regard to Mr. Nagler's comment on the lack of agreement between Knoop and Vickers numbers, there seems to be very little reason to expect them to be the same for all materials. Probably the tests do not measure identically the same physical properties.

Mrs. Brodie and Mr. Smoluchowski are to be congratulated on a very ingenious approach to the elastic recovery measurement.

The author has tested some electrolytically polished specimens sent by Mr. R. H. Heyer of American Rolling Mill Company. One sample of S.A.E. 4330 steel heat treated to about 475 Brinell showed the characteristic increase in Knoop number at low loads. The other samples were quite soft material and showed, as predicted, only a slight increase in Knoop number at low loads.

SHOT FOR METAL PEENING

BY OSCAR E. HARDER AND JAMES T. GOW

Abstract

The materials which are known to have been used in metal peening include chilled iron shot, of the type used in metal cleaning, malleableized chilled iron, and a special heat treated shot. Generally, chilled iron is too brittle and completely malleableized iron is too soft. The heat treated shot is subjected to heating to decompose the massive carbides and cooled so as to produce different hardnesses in the range of 200 to over 500 Brinell equivalent, or may be quenched to a martensitic structure and then tempered to a desired hardness.

Typical chemical compositions and hardness values are given, and photomicrographs illustrative of the various materials are included.

The heat treated shot has improved resistance to fracturing in service and can be produced in hardness ranges suitable for the majority of metal peening applications but may find limitations with materials of high hardness such as about 50 to 55 Rockwell C and above, because as the hardness of the shot is increased into this range there is a loss in toughness. Steel shot is not known to be available and burnishing balls and ball bearings are costly.

THE increase in interest in improving the fatigue endurance of metals and alloys by shot peening has served to focus attention on the shot used. Interest in the shot is principally along the lines of what material has been used, what materials are now available, and also what materials could be made available: first, at a reasonable cost, and second, at an increased cost but yet at a price commensurate with the benefits to be obtained by metal peening.

The shot which has been most readily available in quantity and at moderate cost has been a chilled iron, although the product is frequently referred to in the commercial trade as steel shot. It is not of the typical analysis used in the production of white iron castings

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. Of the authors, Oscar E. Harder is assistant director and James T. Gow is assistant supervisor, Battelle Memorial Institute, Columbus, Ohio. Manuscript received June 1, 1944.

for use as such or in the production of white iron castings which are to be malleableized. The analysis is more nearly what is known in industry as gray iron, but because of the rapid cooling in a shotting process, the product has a white iron structure in spite of the fact that the composition is such that in sand or chilled castings the structure would be gray, or at least mottled. Shot which we have examined show an entirely white iron structure with cementite and austenite or martensite as the constituents. The microstructure typical of chilled iron shot is shown in Fig. 1, which is at a magnification of $\times 2500$. The white areas are cementite, while the dark areas are austenite-martensite. All specimens were etched in 4 per cent nital.

The composition of chilled iron shot is usually in the following range: carbon 3.20 to 3.60 per cent, manganese about 0.30 to 0.50 per cent, silicon about 1.40 to 2.2 per cent, phosphorus about 0.30 to 0.60 per cent, and sulphur about 0.07 to 0.17 per cent. The composition is a function of the stock used in the charge to the cupola and the temperature and rate of melting. When the charge is made up of more or less miscellaneous cast iron scrap, that is reflected in the analysis, and the same is true as the amount of steel or pig iron of different types is varied in the charge. In addition to the above-mentioned composition, shot will frequently carry small amounts of the alloying metals which are used in cast iron, and these may include copper, nickel, chromium, molybdenum, and vanadium. As a rule, however, the amount of these metals in the shot will rarely exceed a total of 0.10 to 0.20 per cent, with the amounts of the individual metals decreasing to a trace in the order mentioned above.

There has been definite use of copper added to the charge, most frequently in the amount of about 0.25 to 1 per cent, and numerous tests have shown that copper improved somewhat the resistance of shot to fracturing in repeated hammer tests.¹ Certain patent literature has disclosed and claimed the use of combinations of the metals, chromium, nickel, molybdenum, and vanadium, with the metals being used in combinations of 2, 3, and 4 of these metals. No information is available to the writers as to the extent to which shot conforming to these patents has been produced or as to the beneficial effects resulting from such intentional alloy additions.

Grit which is used in metal cleaning is made mostly from the chilled iron product by crushing. There is, however, on the market a grit which is made from high-carbon steel plate, which, according

¹U. S. Patent 2,218,107—October 15, 1940.

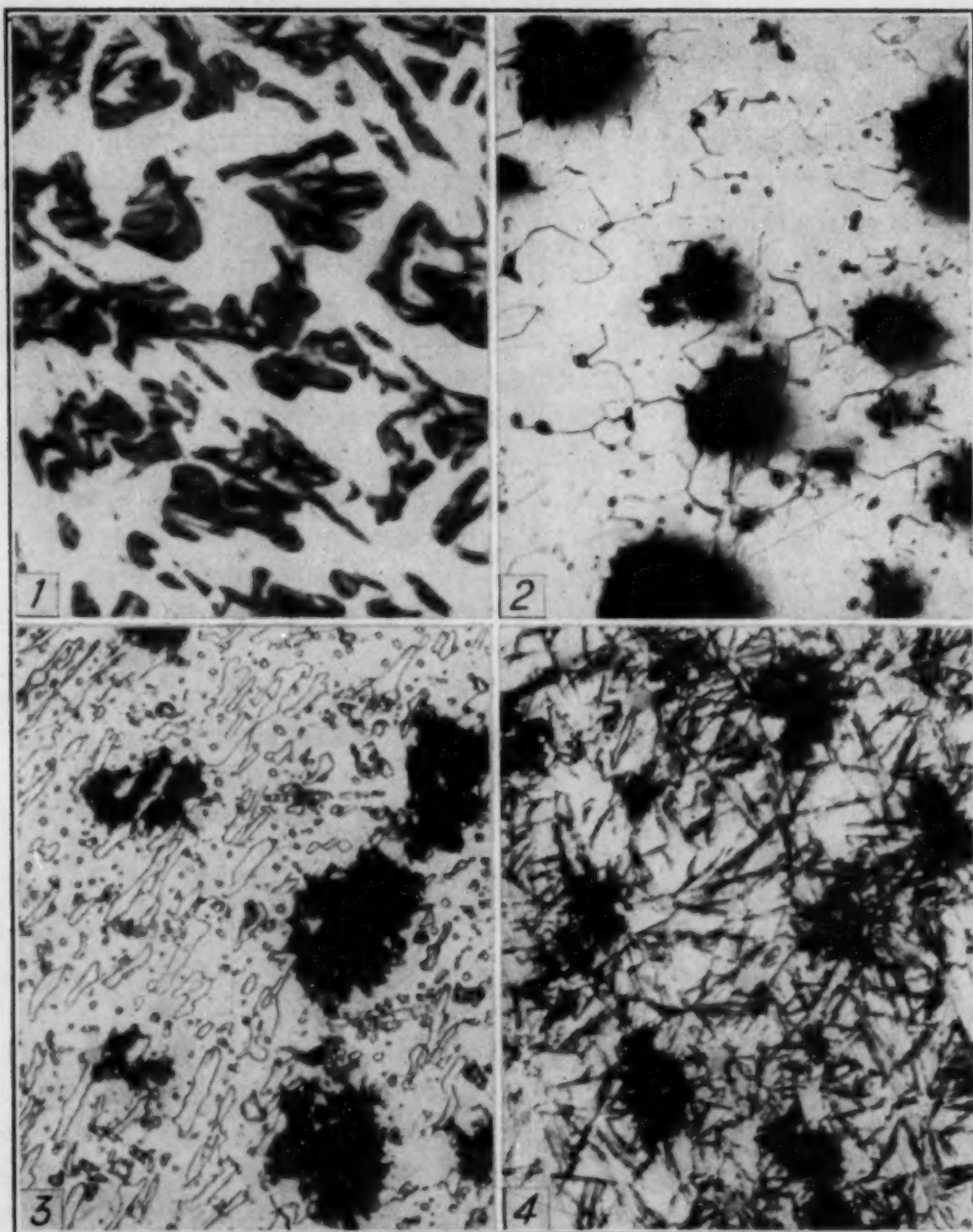


Fig. 1—Microstructure of Chilled Iron Shot. $\times 2500$. Etched in 4 Per Cent Nital.

Fig. 2—Microstructure of Completely Malleableized Chilled Iron Shot. $\times 1000$. Etched in 4 Per Cent Nital.

Fig. 3—Microstructure of Partially Malleableized Chilled Iron Shot Showing Cementite, Ferrite, and Temper Carbon. $\times 1000$. Etched in 4 Per Cent Nital.

Fig. 4—Microstructure of Malleableized and Quench Chilled Iron Shot Showing Martensite and Temper Carbon. $\times 1000$. Etched in 4 Per Cent Nital.

to a patent disclosure, is heated to a high temperature, quenched, and then crushed to grit. This, however, represents a relatively small tonnage of the total amount of grit produced, and grit is not con-

sidered usable for metal peening, since it scratches rather than peens.

The chilled iron shot as normally produced for use in metal cleaning has a hardness of around 800 Vickers and may run as high as about 900. This hardness is well in excess of the hardness of metal parts which it is desired to improve by peening. However, this chilled iron shot is far from being satisfactory for metal peening because of its brittleness and its tendency to fracture or explode, as it is termed in the trade, when impacted against a hard surface. Fractured shot are quite effective for metal cleaning, providing the particles do not become too small in size, but these shot fragments with sharp corners tend to cut the metal and would, therefore, not serve the purpose intended in metal peening. The harmful effects from fractured shot would be reduced somewhat if the system were equipped to remove the fractured particles, but this has not always been done. In spite of the objections mentioned above, this material has been used in most of the work in shot peening. However, two other materials have been used to some extent.

One of these materials is a completely, or almost completely, malleableized shot of the usual analysis, while the other is treated by a process which controls the degree of malleabilization and subjects the shot to a quenching operation and may further subject it to a tempering operation to develop a desired hardness. The malleableized shot, if the process is carried to completion, will be quite low in hardness, with values corresponding roughly from 150 to 200 Brinell. Obviously, shot of such hardness are quite limited in application for metal peening because most of the steels to be peened will have a hardness in excess of 200 Brinell, and it is, therefore, evident that relatively little, if any, improvement could be effected by impacting with shot of lower hardness. Furthermore, the shot of so low a hardness is deformed in service and no longer is so round as seems desirable for peening.

The second product² mentioned above is made by subjecting shot to heating at temperatures in the range of about 1350 to 1600 degrees Fahr. for a time sufficient to bring about the desired degree of decomposition of the massive cementite in the original chilled iron and then cooling at a rate to produce the desired hardness. The rate of decomposition of the cementite increases with increase in treating temperature and, for the usual analysis, is quite rapid ($\frac{1}{2}$ to 1 hour) at 1600 degrees Fahr. (870 degrees Cent.) and can be effected at

²U. S. Patent 2,184,926—December 26, 1939.

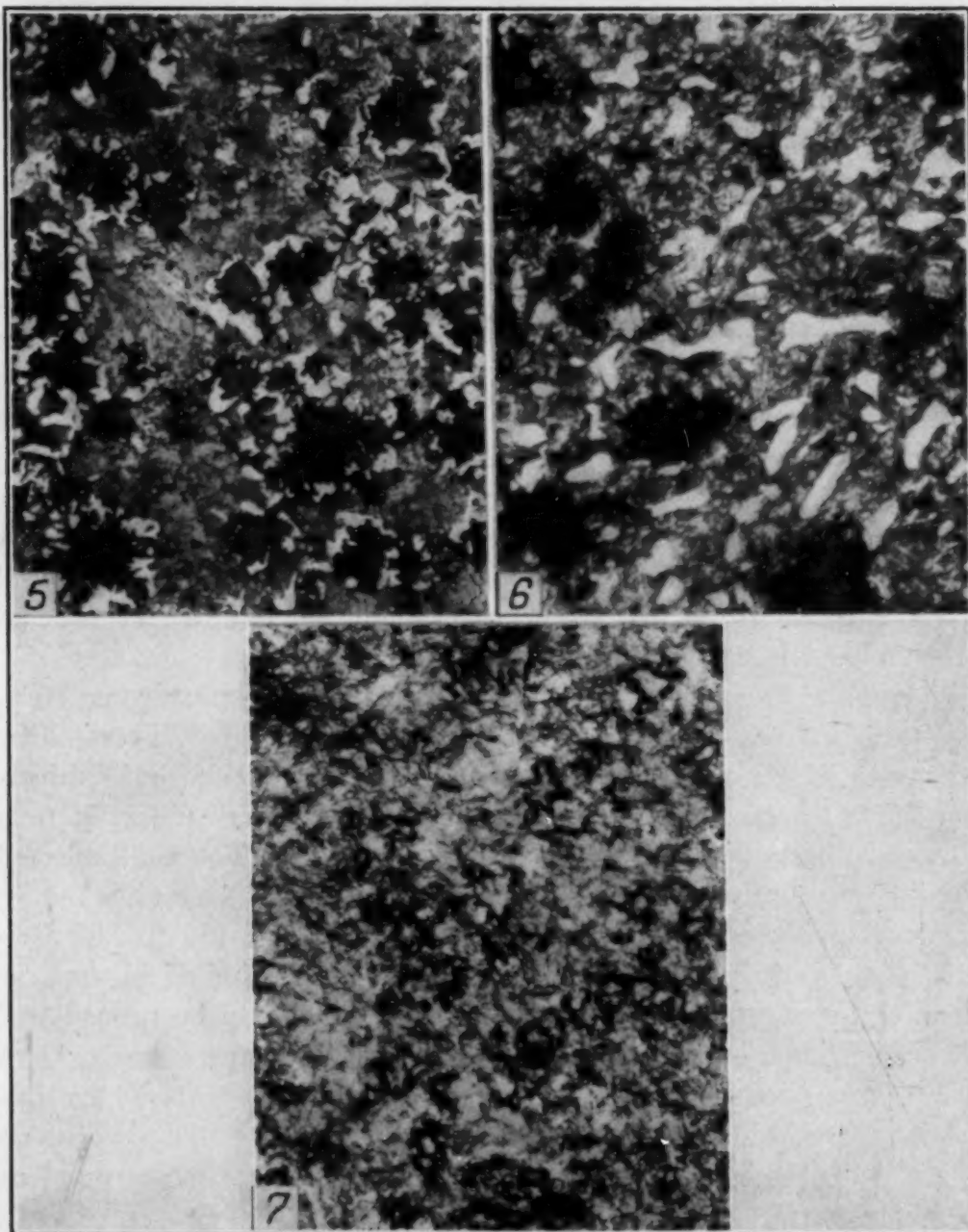


Fig. 5—Microstructure of Malleableized Chilled Iron Cooled to Form Ferrite and Pearlite. $\times 500$. Etched in 4 Per Cent Nital.

Fig. 6—Microstructure of Partially Malleableized Chilled Iron Showing Residual Carbides, Martensite (Troostite?), and Temper Carbon. $\times 1000$. Etched in 4 Per Cent Nital.

Fig. 7—Microstructure of Quenched and Tempered Chilled Iron Shot. $\times 500$. Etched in 4 Per Cent Nital.

1350 degrees Fahr. (730 degrees Cent.) in 3 to 4 hours.

The structure produced is one of temper carbon distributed in the matrix, and the character of the matrix is a function of the tem-

perature from which it is cooled and the rate of cooling. It may be largely martensite or it may be entirely pearlitic, and if the cooling is arrested at the proper temperature for completion of the malleableizing operation, the structure may be largely ferrite and temper carbon. With a drastic quench, hardnesses of the order of 500 to 600 Brinell equivalent are produced, and then the hardness can be reduced to desired lower values by well-known tempering treatments. Thus, this method of treatment is capable of producing shot with hardness easily within the range of 200 to 500 Brinell equivalent with the structure principally that of steel but with temper carbon distributed through it.

Typical microstructures of shot of originally chilled iron, but after various heat treatments, are shown in Figs. 2 to 7. Fig. 2 shows the microstructure of the completely malleableized product in which the structure is temper carbon in a matrix of ferrite.

Fig. 3 shows a specimen in which the malleableizing process was not completed, and, as a result, the structure consists of cementite, ferrite, and temper carbon. This specimen was slowly cooled.

Fig. 4 represents a specimen in which the massive carbides were completely removed in the heat treating process, but there remained enough carbon in solution to produce a martensitic structure on quenching. Thus, the structure is martensite and temper carbon and the hardness at least 500 Brinell equivalent.

Fig. 5 shows the microstructure of a specimen which was heated to decompose the carbides and cooled rather slowly so that there was some separation of ferrite before the eutectoid temperature was reached and pearlite was formed.

Fig. 6 shows a typical microstructure of a product which has not been heated long enough for complete malleabilization, but the massive carbides are broken up so as to materially increase the toughness. This represents a relatively hard shot.

Fig. 7 shows a microstructure which is rather typical of one form of process in which the shot is quenched to a temperature of the order of 400 to 500 degrees Fahr. (205 to 260 degrees Cent.) and then permitted to cool slowly so that a quenched and tempered structure is obtained.

The toughness of the shot described above is greatly superior to that of the ordinary chilled white iron of commerce but is probably somewhat inferior to steel, although that relation has not been determined by the authors. As would be expected, the toughness of the

shot decreases somewhat over the higher hardness range, but this trend is not noticeable up to a hardness of around 400 Brinell equivalent, and it is possible to deform the shot substantially by hand hammering up to 450 Brinell equivalent, without fracturing.

It is indicated by those working on shot peening that shot in the whole size range from about 0.01 to 0.10 inch in diameter may be required, but the principal demand will probably be in the range of 0.015 to 0.030 inch. It is possible to produce all of these sizes in the shotting process, but it may be difficult to produce the desired amounts of some of the sizes, particularly if there should be a large demand for shot in the diameter range of 0.015 to 0.020, or 0.030 to 0.040, for example. An excessive demand for particular sizes would mean that the shot would have to be screened before the heat treatment and the sizes not in demand for metal peening diverted to other applications. The nature of the shot peening process makes it desirable to have the shot of uniform diameter within close tolerances and supplied to a specified diameter. This has not always been done but is possible by suitable screening processes.

This heat treated malleableized shot does not meet all of the specifications being mentioned by users of shot because when the shot is made to have a hardness in excess of some of the parts to be treated, it becomes somewhat brittle and will undoubtedly produce some fracturing in service. However, these specifications will probably not be entirely met should they go to a steel shot which would be much more expensive.

So far as is known to the authors, steel shot is not now being produced by a shotting process comparable to that used in the production of chilled iron shot. However, at one time a plant was melting high-carbon steel in an electric furnace and shotting it to produce steel shot for use in the granite industry. That plant is thought to have been out of use for the past several years, and the company has returned to the use of chilled iron shot. It seems possible to produce steel shot, but the cost would undoubtedly be much higher than that for white iron because of the higher temperature required to produce steel of sufficient fluidity for shotting. Steel shot of the type used as burnishing balls and ball bearings might be considered, but it is thought that the cost would be prohibitive. Definite quotations on such shot are not available, but one estimate on the price of burnishing balls in the size of $\frac{1}{16}$ to $\frac{3}{32}$ inch diameter indicated prices of the order of fifty to seventy-five cents per pound,

i. e., \$1000.00 to \$1500.00 per ton.³ Doubtless the price could be reduced on large quantity production, but it would still seem to be prohibitive for most shot peening applications. Finally, even steel shot or balls naturally show a loss in resistance to fracture as the hardness is increased.

Battelle Memorial Institute has been engaged during the past several years in a research on the improvement of shot for the Pittsburgh Crushed Steel Company and its subsidiaries, the Globe Steel Abrasive Company, the American Steel Abrasives Company, and the Steel Shot and Grit Company, Inc. The Globe Steel Abrasive Company has been producing the heat treated shot described above for the past 3 or 4 years, and it is understood that plans are under way to materially increase the capacity to produce this type of shot. The authors are glad to acknowledge their indebtedness to the Pittsburgh Crushed Steel Company and its subsidiaries and to Battelle Memorial Institute for the privilege of publishing this paper.

DISCUSSION

Written Discussion: By J. H. Frye, Colonel, Ordnance Department, War Department, Washington, D. C.

The authors have selected a timely subject and approached the problem in a realistic manner.

They have capably dealt with the shotting method as applied to extremely fluid cast iron and the benefits of heat treating the chilled shot. But no small part of the value of their paper lies in its thought-provoking aspects.

Shot peening is most beneficial on extremely hard materials, largely because there is not much else to be done in such instances, but less spectacular improvement can be obtained with reasonably low hardness values. There would seem to be a difference in shot requirements for peening materials ranging from 180 to 600 Brinell. Comparative data on the life of shot made from chilled iron, chilled iron heat treated, and hardened steel shot over this range in hardness would be interesting. Of greater value would be data on the variations of the peening effectiveness of the 3 types of shot over this hardness range, both with and without elimination of broken shot.

I would also like to raise this question. Assuming efficient continuous separation of fractured shot, is a reasonable number of fractures important in the result, exclusive of the economics of shot life?

The authors' comment on the availability and price of hardened steel shot is applicable as of today. But if later developments show an economic need for a ball bearing type of shot in quantities the American Industry will produce

³The current price for chilled iron shot in carload lots is about \$63.00 per ton, while the heat treated shot is about fifty per cent higher.

them. Cost per pound is frequently a poor criterion of value, as demonstrated by the fact that tungsten carbide cutting tools sell for \$30,000 per ton, as compared with high speed steel cutting tools at \$1200 per ton.

Written Discussion: By Maurice Olley, British Army Staff, A.F.V. Branch, Detroit.

I understand that malleable shot have been useful for some applications, notably the peening of track pins, in which they reduced the wastage of shot due to fracture to less than half. It appears, therefore, that for certain applications on parts of low hardness, malleable shot might be worth considering.

It is evident, however, that they do not meet the conditions required for shot peening in its principal applications. The chief aim of shot peening concerns the harder materials. In fact, one aim of peening is to use harder materials than are at present advisable, while obviating, by the peening process, the brittle fatigue fractures which would normally be liable to occur.

Every shot is a hammer head. Hammer heads in cast or malleable iron are unsuccessful; they are not sold by the better hardware stores. When we fully realize the aim and object of the peening process, we shall not attempt to use for peening anything less permanent than we should use for a peening hammer. The first cost will be immaterial, provided the shot does not fracture.

Written Discussion: By W. L. R. Steele, chief engineer, Coil Spring Division, Eaton Manufacturing Co., Detroit.

In the last paragraph of page 395, the authors call attention to the lack of existing data on the relation between white iron shot and steel shot toughness. We have some data on this relation.

When the part to be peened has a hardness in the range of Rockwell C 40 to 50 and requires that large shot ($\frac{1}{8}$ to $\frac{3}{8}$ inch diameter) be thrown from a 19-inch wheel at or near 2250 revolutions per minute, the breakdown of the commercial white cast iron shot is rapid and severe. For such peening work we have produced a small quantity of shot made from S.A.E. 1065 wire, the balls being 0.070 inch in diameter, oil-quenched and drawn to a 49-51 Rockwell hardness.

We have devised and run an impact breakdown test to compare the steel ball and the water-quenched cast iron ball. The test was run in a standard commercial 19-inch wheel cabinet but without screw, elevator or dust collector connections to avoid loss of sample. The wheel hopper was filled from a bin containing a 50-pound sample. When 2200 revolutions per minute wheel speed was obtained, the sample was released into the wheel which threw the shot vertically downward onto a platform of S.A.E. 9262 having a 444 Brinell hardness number and thence into a hopper below the platform. The shot sample was then removed from the lower hopper and carried to the upper wheel hopper bin and this cycle was repeated until breakdown to an arbitrary screen analysis was obtained. The results of this cycling on the cast iron and on the steel were as shown in the table on the next page.

From these data it appears that the 1065 steel balls when thrown onto 444 Brinell hardness steel parts would give a life of 25 to 30 times that of the water-quenched cast iron in production machines. This particular analysis of carbon steel was not selected because we expected the optimum in life from it

Original Samples					
Retained on this screen (openings)	White Cast Iron	S.A.E. 1065 Steel	White Cast Iron 10 Cycles	Steel 250 Cycles	Steel after 300 Cycles
0.0787 Inch	0.5 Per Cent	0	0	0	0
0.066 Inch	97.6 Per Cent	100 Per Cent	64.5	73	47.2
Small screens total	1.9		35.5	27	52.8

but because it was readily available. The possibilities of greatly increasing this life ratio by using balls made from alloys more suitable for repeated impact loading are being investigated.

Written Discussion: By R. J. Thomas, metallurgist, Plant No. 1, Jacobs Aircraft Engine Company, Pottstown, Pa.

Professor Harder and Mr. Gow are to be commended for presenting data on shot for metal peening. Information pertaining to the structures and hardness has been very meager. The writer investigated the structure of shot a few years ago when it was being used as a material for rock drilling in connection with core drilling. The desirable characteristic of the shot at that time was the fact that it was brittle and would fracture easily, giving sharp cutting edges. Shot which was found to be unsatisfactory had a structure consisting of pearlite and areas of temper carbon where the massive cementite had broken down. The satisfactory shot had a fine dendritic structure of martensite and carbide. The use of this type shot for peening results in considerable fracturing of the shot with resulting high replacement charges.

It is desirable, in addition to knowing the structural composition and hardness of shot, to know the percentage of satisfactory spheres present. For this purpose a macrophotograph of a small quantity representative of the shipment will reveal the general condition.

Fig. A shows a macrophotograph at $\times 20$ of the shot from a sample kit used by a shot vendor. This was listed as No. 30 shot.

Fig. B shows the condition of shot taken from a 100-pound bag received for use as peening shot.

The condition of this shot indicates that it should be screened prior to use for peening. This operation would result in less than 50 per cent recovery of suitable shot.

Fig. C shows the above shot after approximately 50 hours' use. No new shot had been added. The Almen arc height, however, was still within the limits set. A regulated system of new shot additions was arranged.

The above photographs show the need for control of not only the structure and hardness, but also the percentage of usable shot obtained from any given shipment.

Oral Discussion

R. E. CRAMER:⁴ I just want to call attention to the beautiful photomicrographs that are in this paper. And if either one of the authors at the end of

⁴Special research associate professor of engineering materials, University of Illinois, Urbana, Ill.

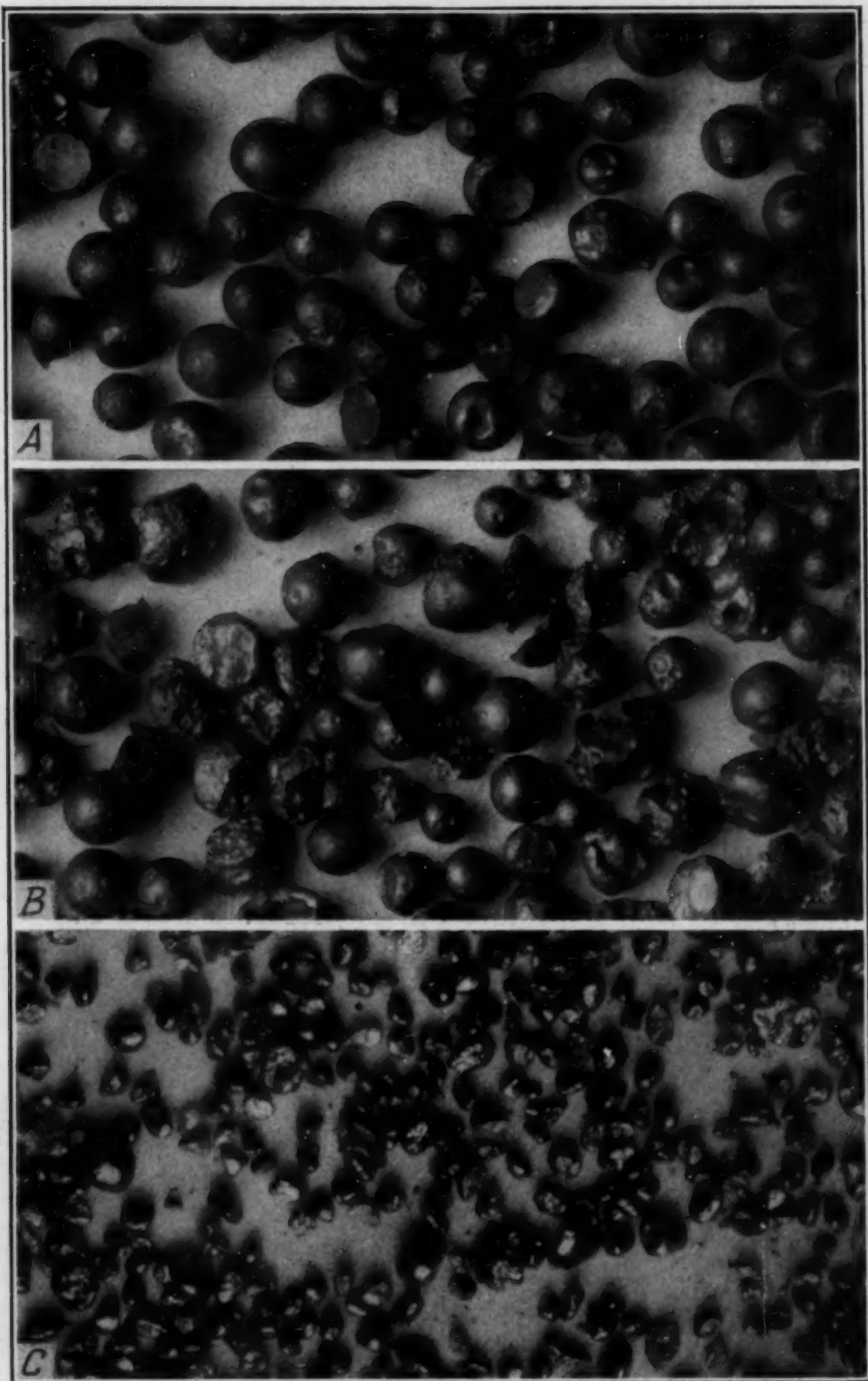


Fig. A—Condition of No. 30 Shot from Sample Kit. $\times 20$.

Fig. B—Condition of Shot from 100-Pound Bag. $\times 20$.

Fig. C—Condition of Shot After Approximately 50 Hours. $\times 20$.

the discussion would give us a little indication as to their method of polishing and etching, and more about the magnification, I think some of us could profit by that. Also, I wish the metallurgical schools could turn out metallurgists who could take pictures like that. I cannot do it. I cannot even hire a man who can do it.

Authors' Reply

We appreciate the valuable contributions made by the discussers of our paper.

Complete answers to the important questions by Colonel Frye are not available now. Mr. Steele has reported comparative fracture data for chilled iron and steel shot of relatively large size, 0.070-inch diameter. It is to be expected that shot of smaller diameter, say, 0.010 to 0.020 inch, would show the chilled iron shot in somewhat less unfavorable ratio. Mr. Olley has reported the breakdown of heat treated iron shot as less than half of that of chilled iron in a particular application. Mr. Thomas has reported that chilled iron shot after 50 hours, with no new shot added, still met the Almen arc height test. While the breakdown of chilled iron is excessive, this is not necessarily so expensive because fractured shot are readily usable for metal cleaning, and most plants which are doing metal peening are also doing metal cleaning.

In reply to Mr. Olley, it is suggested that heat treated iron shot do not appear to be limited to "low hardness" parts, but are or can be made to apply up to hardnesses of 400 to 500 Brinell. However, again it is anticipated that steel shot will be superior in performance but much more expensive, particularly in the smaller sizes.

Mr. Steele has contributed valuable information in his discussion. Since his comparison was made between "white cast-iron" shot and "quenched and drawn" steel shot, it seems that the heat treated iron should show up more favorably than the white iron, and such data would be of interest. It will be evident that the production of steel balls in the range of $\frac{1}{16}$ to $\frac{1}{8}$ inch diameter (0.0625 to 0.09375 inch) is an easier job than the production of the smaller diameters, such as 0.010 to 0.020 inch, which are required for many of the shot peening applications. The results of Mr. Steele's researches, which are in progress, should be of great interest.

The discussion of Mr. Thomas is highly interesting. He has mentioned two uses of chilled iron shot in which the desired behaviors of the shot are in marked contrast. He has shown that manufacturers of chilled iron shot have not been doing an adequate job in screening chilled iron shot for use in metal peening, and it is understood that more attention is being given to sizing shot, at least by some manufacturers.

Professor Cramer's complimentary oral remarks are appreciated. The paper gives the etching reagent used in preparing the microstructures.

SEGREGATION OF MOLYBDENUM IN PHOSPHORUS-BEARING ALLOYED GRAY CAST IRON

BY F. B. ROTE AND W. P. WOOD

Abstract

Phosphorus is shown to be soluble in the particular irons studied to the extent of about 0.12 per cent. Any additional phosphorus occurs in a complex eutectic which, if molybdenum is also present in the iron, contains 1.3 units of molybdenum for each unit of phosphorus. The characteristic high strength acicular microstructure is changed to a pearlitic structure as the phosphorus is increased sufficiently above the solubility limit. Attempts to control the structure of high phosphorus irons by compensatory molybdenum additions are only partially successful, due to a pearlitic envelope surrounding the phosphide eutectic.

The segregation of molybdenum takes place on cooling from the iron-iron carbide eutectic to the iron-iron phosphide eutectic. A reversal of the reaction does not occur on reheating to the iron-iron carbide eutectic temperature.

INTRODUCTION

BECAUSE of the usual intricacy or great bulk of gray iron castings, it is generally not advisable to heat treat them by the conventional quench and draw to obtain certain desirable high strength microstructures. Consequently, modern foundry practice now calls for the introduction of alloying elements to secure mechanical properties greater than those obtainable in ordinary low carbon, unalloyed irons.

The structure of the highest test irons is acicular, of a type intermediate between pearlite and martensite. This structure is se-

This paper is based on a thesis submitted by F. B. Rote in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the Horace H. Rackham School of Graduate Studies at the University of Michigan.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. Of the authors, F. B. Rote is research metallurgist, Wyman-Gordon Company, Worcester, Massachusetts, and W. P. Wood is professor of chemical and metallurgical engineering, University of Michigan, Ann Arbor, Mich. Manuscript received June 17, 1944.

cured primarily by the addition of molybdenum, which forms a "bay" in the S-curve of gray iron as described by Flinn and co-workers^{1,2}, although it is the usual practice to supplement the molybdenum alloy with nickel, copper, or manganese. One widely used combination is nickel and molybdenum in balanced proportions to obtain a favorable graphite structure and an acicular matrix.

In previous research work by the writers it was found that a number of castings made under ordinary foundry conditions were pearlitic, although they were alloyed to an extent which should render them acicular on the basis of the available information. Naturally, the mechanical properties were lower than anticipated, and the castings were unable to meet the high strength specifications. A scrutiny of the entire chemistry of the castings indicated that in only one significant particular did they vary from the irons for which considerable data were available, and that was in the higher phosphorus content. A survey of the literature showed then that no recognition had been given in the previous investigations of irons of the acicular type to the possible effects of the phosphorus on the structure or properties.

An investigation was, therefore, conducted to determine the influence of phosphorus on the solidification and austenite transformation characteristics of gray cast iron, both plain and alloyed with nickel and molybdenum. The results of one phase of this investigation are reported in the present paper.

EXPERIMENTAL PROCEDURE

The base analysis of the irons made for this investigation was:

	Per Cent
Total carbon	2.65
Silicon	2.25
Manganese	1.20
Sulphur	0.03
Phosphorus	0.016 to 1.83

Some of the heats were poured without additions, while others were alloyed with 1.75 per cent nickel and 0.75 per cent molybdenum.

The iron was made in 30-pound melts in an Ajax induction furnace from charges consisting of wash metal, low carbon steel punch-

¹R. A. Flinn and D. J. Reese, *Transactions, American Foundrymen's Association*, preprint No. 4, 1941.

²R. A. Flinn, Morris Cohen and John Chipman, "The Acicular Structure in Nickel-Molybdenum Cast Irons," *TRANSACTIONS, American Society for Metals*, Vol. 30, 1942, p. 1255.

ings, ferro-alloys, nickel, and coke. All melts were heated to 2750 degrees Fahr. (1510 degrees Cent.) and poured at 2650 degrees Fahr. (1455 degrees Cent.), as indicated by an optical pyrometer.

Phosphorus was added to the irons in the form of ferrophosphorus containing 24 per cent phosphorus, and the furnace charges were adjusted to compensate for changes in the quantity of alloy charged for low or high phosphorus contents by changes in the quantity of steel punchings charged. A typical charge for an alloyed melt was as follows:

Heat No. 5 (0.58 Per Cent P)	
	Grams
Wash metal	8,100
Steel punchings	4,225
Nickel	240
Ferro-60-molybdenum	175
Ferro-85-silicon	295
Ferro-24-phosphorus	375
Petroleum coke	130
Total	13,540

All alloys were charged with the steel and part of the wash metal at the beginning of each heat to reduce the chance of inoculation of the melt by ferrosilicon.

The chemical compositions of the melts are shown in Table I.

A number of different castings were made from each heat; however, for the purposes of the present paper only one will be considered, a solid cylinder 3 inches in diameter by 4.5 inches in

Table I
Chemical Compositions of Unalloyed and Nickel-Molybdenum Alloyed Gray Irons

Heat No.	T.C.	C.C.	Si	Mn	Ni	Mo	S	P
1	2.61	0.59	2.24	1.19	1.79	0.77	0.030	0.016
11	2.59	0.61	2.29	1.20*	1.75*	0.75*	0.03*	0.14
8	2.64	0.63	2.29	1.20*	1.75*	0.75*	0.034	0.34
12	2.63	0.67	2.21	1.20*	1.75*	0.75*	0.03*	0.45
5	2.66	0.63	2.23	1.20*	1.75*	0.75*	0.03*	0.58
14	2.64	0.65	2.26	1.22	1.77	0.76	0.029	0.77
7	2.61	0.67	2.31	1.20*	1.75*	0.75*	0.03*	0.95
2	2.64	0.66	2.22	1.20*	1.75*	0.75*	0.03*	1.25
10	2.63	0.69	2.25	1.20*	1.75*	0.75*	0.03*	1.83
31	2.65*	2.25*	1.22	0.032	0.018
32	2.66	2.21	1.20*	0.03*	0.37
33	2.65*	2.25*	1.20*	0.03*	0.93
34	2.68	2.25*	1.20*	0.03*	1.80*

Late Addition—None

* Calculated analysis

length. This casting was made in an open top core mold, and was poured directly from the induction furnace.

After the casting was poured and skimmed, a calibrated platinum thermocouple sheathed in a thin wall quartz tube and centered by means of a transite disk was inserted to the geometric center of the molten iron. As soon as the thermocouple reached the metal temperature, the first temperature reading was made on a Leeds and

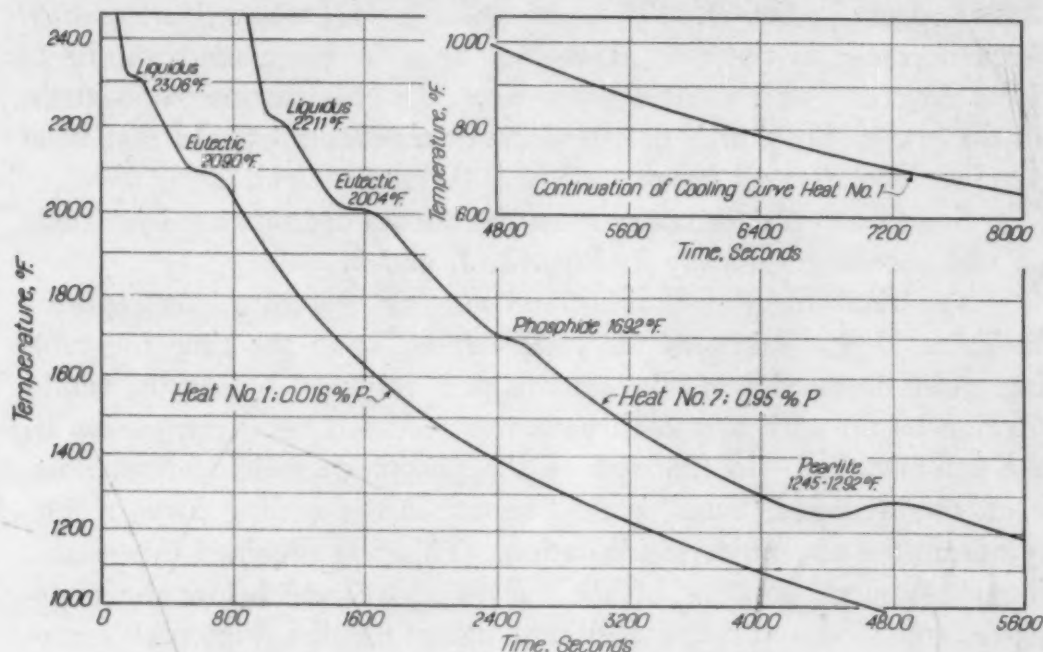


Fig. 1—Cooling Curves for 3-Inch Castings of Heats Nos. 1 and 7 (Base Analysis —2.65 Total Carbon, 2.25 Silicon, 1.75 Nickel, 0.75 Molybdenum).

Northrup portable precision potentiometer. The temperature at this time was usually about 2450 degrees Fahr. (1345 degrees Cent.). Thereafter, readings were taken each 10 seconds until the castings had cooled to below the pearlite transformation temperature in those irons which underwent a pearlite transformation, and to 400 degrees Fahr. (205 degrees Cent.) in the acicular irons.

COOLING CURVE DATA

Cooling curves were obtained for all the castings, but for demonstration, only those for alloyed heats numbers 1 and 7 with 0.016 and 0.95 per cent phosphorus, respectively, will be discussed. These are shown in Fig. 1.

Heat 1 began to solidify at 2306 degrees Fahr. (1265 degrees Cent.), as indicated by the first "halt" in the cooling curve, then

cooled at a fairly constant rate to the eutectic temperature of 2090 degrees Fahr. (1145 degrees Cent.) and completed its solidification. No further allotropic transformation was observed, however, upon further cooling of the casting.

Heat 7 with 0.95 per cent phosphorus reached the liquidus at 2211 degrees Fahr. (1210 degrees Cent.), then cooled with the continued solidification of austenite to the eutectic at 2004 degrees Fahr. (1095 degrees Cent.). The phosphide solidification was marked by a decrease in the rate of cooling at an average temperature of 1692 degrees Fahr. (920 degrees Cent.). This casting, in contrast to the lower phosphorus one, underwent a pearlite transformation at 1245 to 1292 degrees Fahr. (675 to 700 degrees Cent.).

Solidification data taken from the curves are tabulated in Table II and shown graphically in Figs. 2, 3, and 4.

The time for the liquidus and eutectic solidifications reported in Table II was taken as the total elapsed from the time the cooling curve deviated from the smooth path followed before the solidification began until a smooth path was resumed upon completion of the solidification. In the case of the phosphide solidification, however, the tabulated "time" is the "offset" in the cooling curve resulting from the phosphide solidification. This was obtained by extending the smooth portions of the curves above and below the phosphide solidification range and measuring the time interval represented by the distance between the curves at the average solidification temperature.

Table II
Solidification Temperatures in Centers of 3-Inch Diameter Castings

Heat No.	Per Cent P	Liquidus		Eutectic Temperature, °F.			Phosphide	
		Temp. °F.	Time Secs.	Min.	Max.	Ave.	Time Sec.	Temp. °F. Time (2) Sec.
1	0.016	2306	70	2086	2090	2088	210 None
11	0.14	2285	50	2067	2070	2069	200 None
8	0.34	2280	60	2055	2058	2056	230	1652 35
12	0.45	2261	80	2040	2045	2043	200	1669 57
5	0.58	2231	60	2032	2036	2034	230	1667 83
14	0.77	2226	70	2021	2022	2022	210	1673 100
7	0.95	2211	80	2000	2004	2002	220	1692 160
2	1.25	2170	70	1985	1990	1986	230	1712 180
10	1.83	2096	70	1992	1995	1993	210	1727 280
31(1)	0.018	2350	60	2055	2062	2058	100 None
32	0.37	2306	50	2012	2017	2015	130	1727 18
33	0.93	2252	50	1982	1982	1982	140	1735 57
34	1.80*	2155	60	1968	1972	1970	140	1753 121

Calculated Analysis

(1) Heats 31 to 34 were unalloyed, all others contained nominally 1.75 per cent nickel and 0.75 per cent molybdenum.

(2) Offset in cooling curve.

Since in the lower phosphorus irons the rate of phosphide eutectic solidification was very slow, while in the higher phosphorus irons it was considerably more rapid, depending on the phosphorus content, the total time elapsed during the solidification was about the same for all phosphorus levels. It was necessary, therefore, to utilize

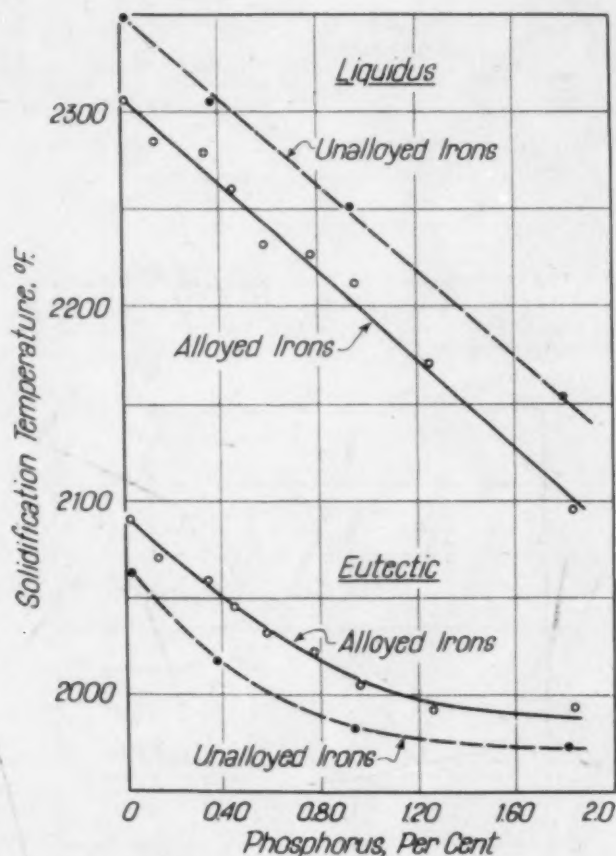


Fig. 2—Influence of Phosphorus on the Solidification Temperatures in 2.65 Total Carbon, 2.25 Silicon Base Gray Irons.

the "offset" procedure to obtain a measure of the effect of phosphorus content on the cooling of the casting.

As shown in Fig. 2, as the phosphorus content of either type iron was increased, the liquidus and eutectic temperatures were lowered. Phosphorus affected both the alloyed and unalloyed irons similarly, since the curves of solidification temperature versus phosphorus contents are essentially parallel. This effect of phosphorus on the solidification temperatures of iron-carbon alloys is well known, and has been disclosed by a number of investigators in the past. Use

will be made, however, of the comparative data for the two types of iron in a later discussion.

Fig. 3 shows the influence of phosphorus on the solidification temperatures of the phosphide eutectic in the unalloyed and nickel-molybdenum alloyed irons. In the plain irons, increasing phos-

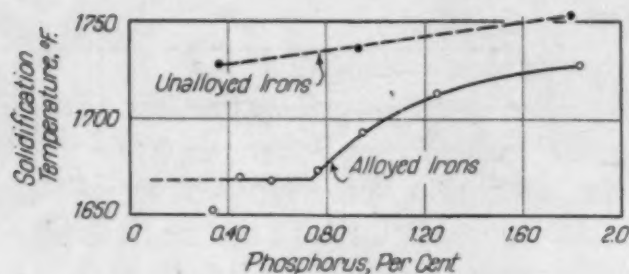


Fig. 3—Influence of Phosphorus on the Temperature of Solidification of the Iron Carbide-Iron Phosphide Eutectic in 2.65 Total Carbon, 2.25 Silicon Base Gray Irons.

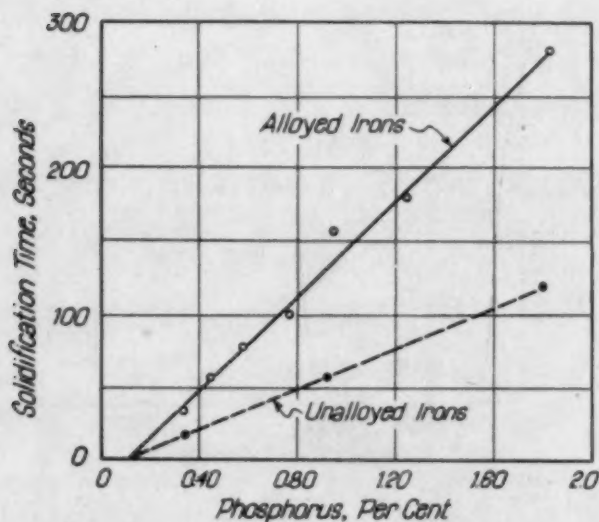


Fig. 4—Influence of Phosphorus on the Time (Offset) Required for the Solidification of the Iron Carbide-Iron Phosphide Eutectic in 2.65 Total Carbon, 2.25 Silicon Base Gray Irons.

phorus content resulted in slightly higher solidification temperatures, probably because of the lower degree of undercooling of the eutectic in larger quantities.

The phosphide solidification temperatures in the alloyed irons were considerably lower than in the plain irons, and the curve obtained in the plot was quite different from that for the plain irons. It is apparent from a comparison of the two curves that the chemical composition of the phosphide in the alloyed iron varied somewhat

with phosphorus content, since "undercooling" cannot reasonably be used to explain the wide differences in temperature observed. This will be discussed in a later section.

Fig. 4 demonstrates in another manner the differences in properties of the two phosphides. The "offset" time required for the solidification of the phosphide in the alloyed irons was about twice as great as in the unalloyed irons. Since the "offset" time is in reality the "time lag" brought into the normal cooling by the phosphide solidification, it is apparent that the quantity of phosphide in the alloyed iron must have been greater or the latent heat of fusion per unit greater than in the unalloyed iron. Either condition indicates a difference in composition.

Extrapolations of the plots of "offset" time versus phosphorus content to low phosphorus content in both types of iron intersect the "zero" time axis at about 0.12 per cent phosphorus. Since the "offset" time is an indication of the disturbance in cooling when the phosphide solidifies, the intersection indicates no disturbance; or no phosphide. This point shows, therefore, the maximum solubility of phosphorus in the particular irons studied, 0.12 per cent.

METALLOGRAPHIC STRUCTURES

As is shown in Fig. 1, no thermal indication of an austenite transformation was observed on cooling the 3-inch casting of heat No. 1. After the eutectic solidification, the cooling progressed smoothly with a gradually diminishing rate. Subsequent metallographic examination disclosed that the austenite transformation product was acicular of a type intermediate between pearlite and martensite. According to Flinn,^{1,2} transformation of austenite to this structure takes place at between 600 and 800 degrees Fahr. (315 and 425 degrees Cent.). This was confirmed by examination of specimens from the acicular irons after isothermal transformation at temperatures between 600 and 1000 degrees Fahr. (315 and 540 degrees Cent.). The transformation rate in this temperature range is very slow which, coupled with the slow natural cooling rate of the 3-inch casting, explains the failure of the cooling data to disclose the transformation.

Heat No. 11 (0.14 per cent phosphorus) behaved similarly. Heat No. 8 (0.34 per cent phosphorus) and all the higher phosphorus alloyed heats underwent partial or complete transformation,

¹Loc. cit.

²Loc. cit.

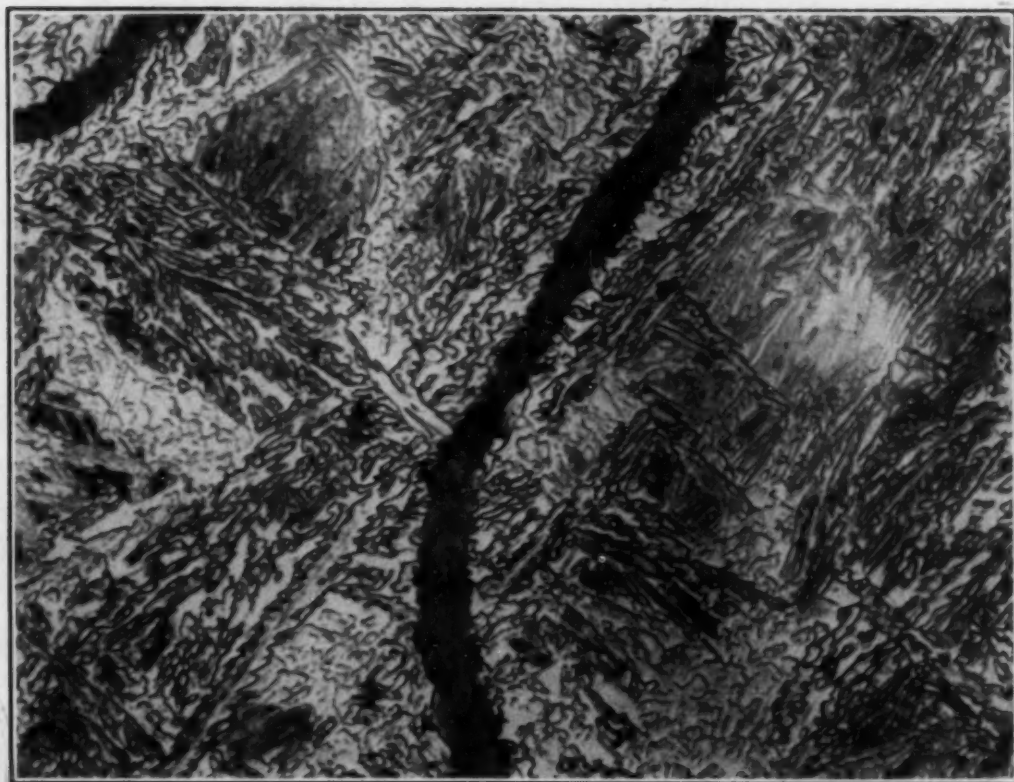
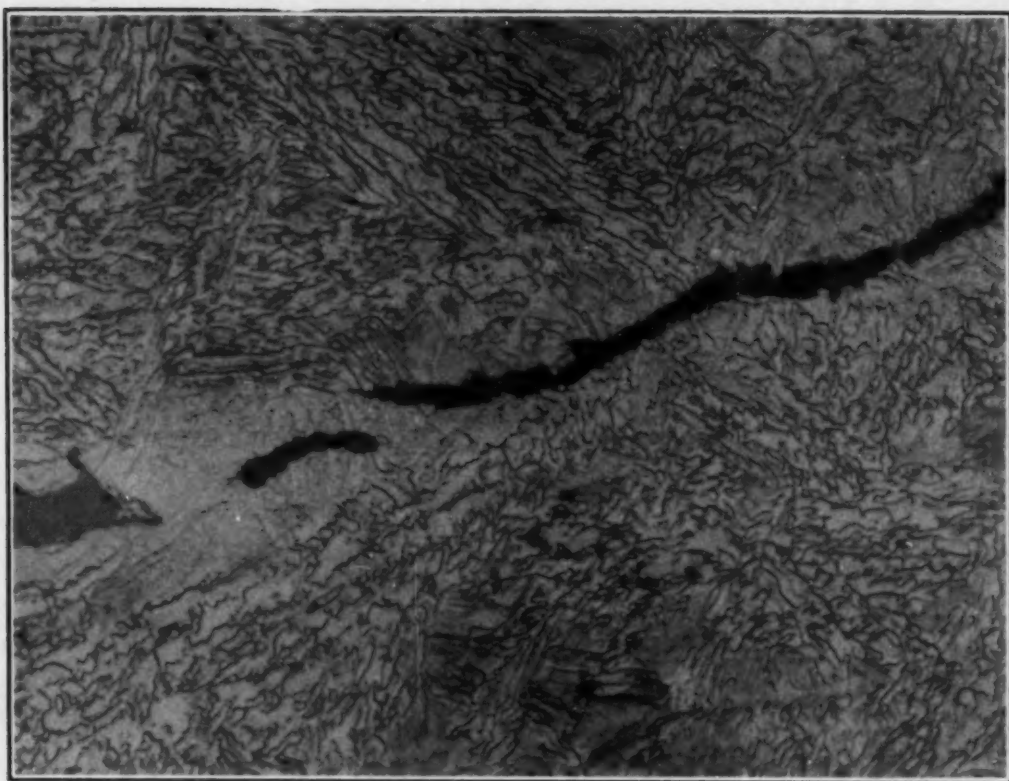


Fig. 5 (a) Upper, (b) Lower—Microstructures at the Centers of 3-Inch Castings of Alloyed Gray Iron. Base Analysis: 2.65 Total Carbon, 2.25 Silicon, 1.20 Manganese, 1.75 Nickel, and 0.75 Molybdenum.
a—0.016 Per Cent Phosphorus—Fully Acicular Structure. $\times 1000$.
b—0.14 Per Cent Phosphorus—Fully Acicular Structure. $\times 1000$.

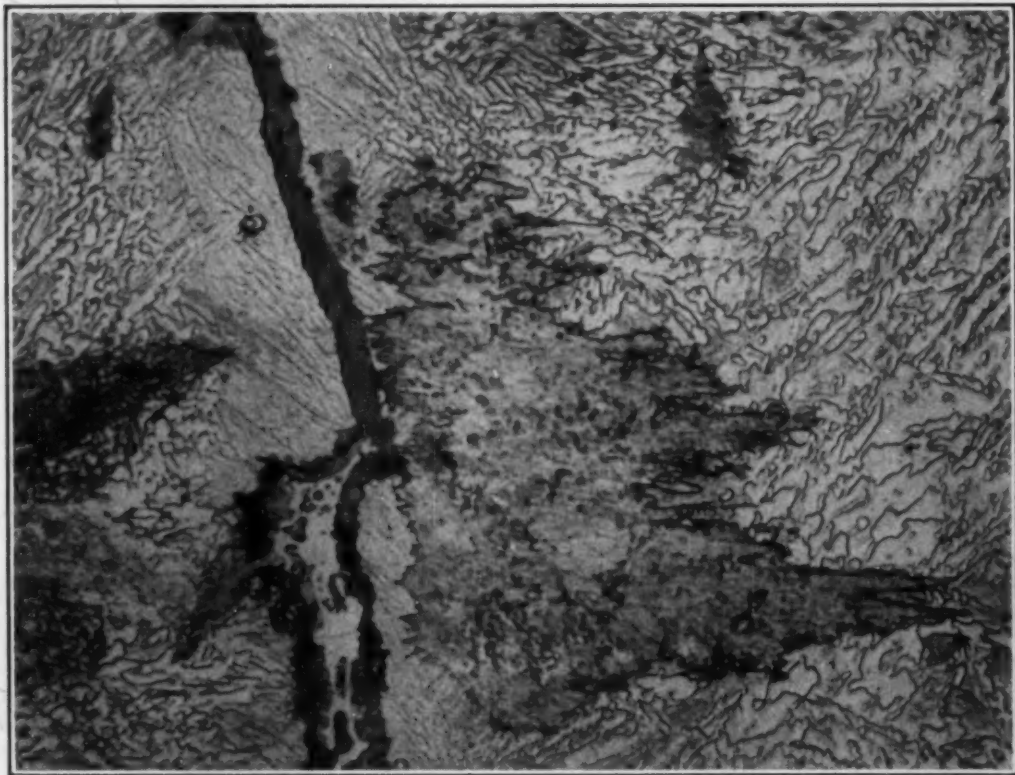
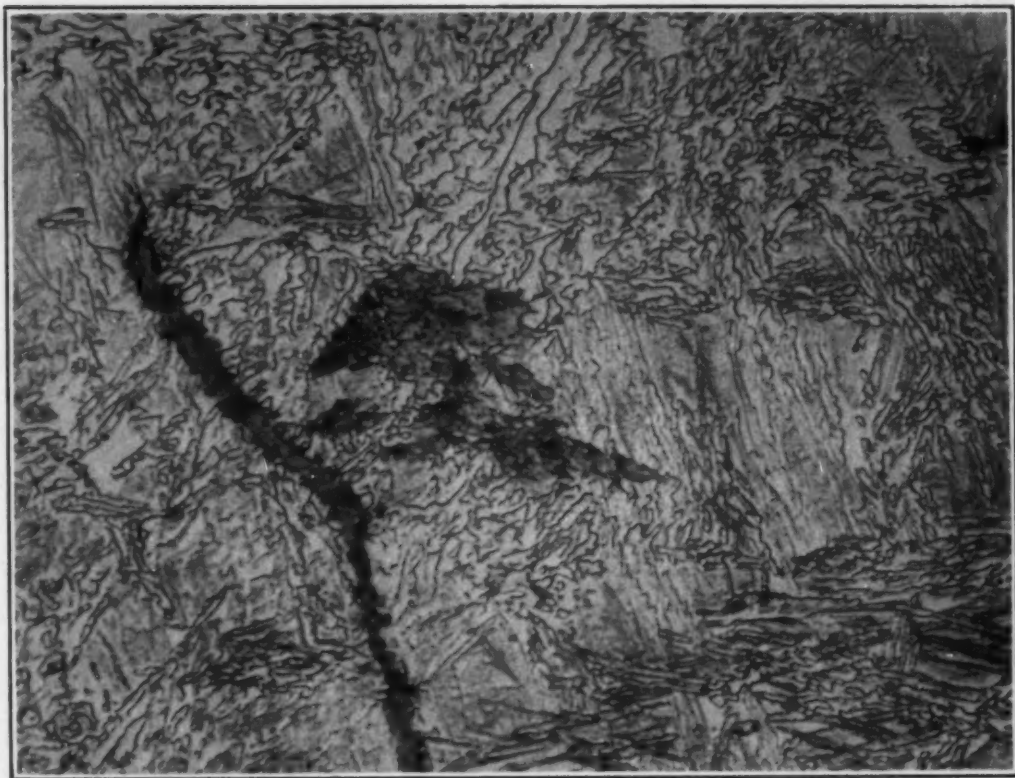


Fig. 5 (c) Upper, (d) Lower—Microstructures at the Centers of 3-Inch Castings of Alloyed Gray Iron. Base Analysis: 2.65 Total Carbon, 2.25 Silicon, 1.20 Manganese, 1.75 Nickel, and 0.75 Molybdenum.
c—0.34 Per Cent Phosphorus—Acicular + 5 Per Cent Pearlite. $\times 1000$.
d—0.45 Per Cent Phosphorus—Acicular + 10 Per Cent Pearlite. $\times 1000$.

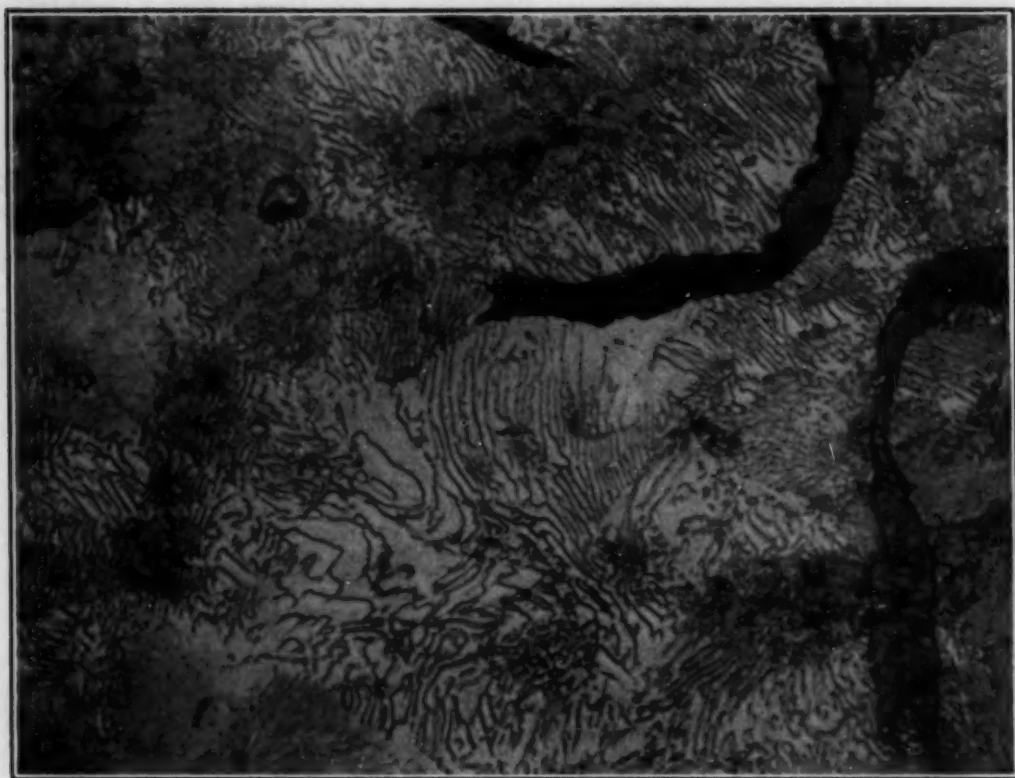


Fig. 5 (e) Upper, (f) Lower—Microstructures at the Centers of 3-Inch Castings of Alloyed Gray Iron. Base Analysis: 2.65 Total Carbon, 2.25 Silicon, 1.20 Manganese, 1.75 Nickel, and 0.75 Molybdenum.

e—0.58 Per Cent Phosphorus—Pearlite + 2 Per Cent Acicular. $\times 1000$.

f—0.77 Per Cent Phosphorus—Fully Pearlitic Structure. $\times 1000$.

as indicated by the cooling curves, at between 1145 and 1292 degrees Fahr. (565 and 700 degrees Cent.), all within the pearlite range. The microstructures of the castings are shown in Fig. 5.

The 0.016 and 0.14 per cent phosphorus heats were fully acicular with the structure predominantly of the intermediate type, but with small quantities of martensite. The 0.34, 0.45, and 0.58 per cent phosphorus heats were of mixed microconstituents; predominantly acicular with a little pearlite in the 0.34 phosphorus heat, and predominantly pearlitic with a little acicular product in the 0.85 per cent phosphorus heat. At higher phosphorus levels the castings were fully pearlitic.

The matrix microstructures of the unalloyed irons were relatively unchanged with varying phosphorus content, except for increases in the quantity of steadite at high phosphorus levels.

The austenite transformation data based on thermal analysis of the alloyed castings during cooling and metallographic examination and comparison with isothermally transformed specimens are presented in Table III.

Table III
Austenite Transformation Temperatures in Centers of 3-Inch Diameter Castings

Heat No.	Per Cent P	Pearlite Transformation Temperature (°F.)			Acicular Transformation Temp. Range*
		Min.	Max.	Ave.	
1	0.016	650-750 and 450-500
11	0.14	650-750 and 450-500
8	0.34	1145	650-750
12	0.45	1200	1225	1212	650-750
5	0.58	1234	1268	1249	800
14	0.77	1247	1276	1261
7	0.97	1245	1292	1268
2	1.25	1254	1281	1269
10	1.83	1258	1292	1275
31	0.018	1333	1336	1335
32	0.37	1365	1372	1369
33	0.93	1365	1371	1368
34	1.80*	1366	1382	1374

* Estimated by comparison of as-cast microstructure with the microstructure of isothermally transformed specimens.

THE PHOSPHIDE EUTECTIC

During the course of the investigation it was noted that small spherical particles were usually formed on the top surfaces of the higher phosphorus castings in the alloyed series. These particles were firmly fixed to the surface and could be dislodged only by fairly sharp blows with a hammer. In order to establish their identity



Fig. 6—Photograph of the Top Face of the 3-Inch Castings from Heat N6. Analysis: 2.65 Total Carbon, 2.25 Silicon, 1.20 Manganese, 1.75 Nickel, 0.75 Molybdenum, and 0.80 Phosphorus. Notice the Small Pellets Which Adhere to the Surface Even After Sand Blasting. $\times 1$.

and to determine the conditions under which these pellets were formed, the 3-inch casting from an alloyed heat with 0.80 per cent phosphorus was carefully skimmed after pouring and observed during its entire solidification and cooling until the pellets were formed.

This casting solidified with a smooth concave top and proceeded to cool until the surface reached about 1750 degrees Fahr. (955 degrees Cent.), as measured with the optical pyrometer. Small beads began to form at this time and continued to increase in size until the casting surface reached about 1600 degrees Fahr. (870 degrees Cent.). Unfortunately no photograph of this particular casting was made. However, another casting of the same analysis and poured at the same time for another purpose, but which was not skimmed, was available. A photograph of the casting, N6, after sand blasting is shown in Fig. 6. The beads adhered tightly even after sand blasting.

The microstructure of one of the pellets from the top of the casting is shown in Fig. 7. It was characteristically eutectic with a structure identical to the phosphide eutectic areas in the higher phosphorus irons. (Compare with Fig. 5f.)

Because of the microstructure of the pellets and temperature at which they were formed, it was obvious that they were composed of the phosphide eutectic. The entire yield from each available casting was, therefore, gathered and analyzed for carbon, silicon, nickel, molybdenum, and phosphorus. The results of the chemical analyses are given in Table IV.

Table IV
Chemical Compositions of Phosphide Eutectics Extruded from the Alloyed Castings

Analysis of Base Iron					Analysis of Eutectic					Heat Number
T.C.	Si	P	Mo*	Ni*	T.C.	Si	P	Mo	Ni	
2.61	2.31	0.95	0.75	1.75	4.11	3.75	1.65	7
2.64	2.22	1.25	0.75	1.75	2.06	1.02	4.27	2.19	1.61	2
2.65*	2.25*	0.95*	0.75	1.75	1.00	4.37	3.96	511
2.65*	2.25*	1.60*	0.75	1.75	1.02	4.48	2.23	1.68	513

* Calculated composition

Upon examination of the data in Table IV, it is apparent that the influence of phosphorus on the structure of the alloyed irons lay in the removal of molybdenum from the matrix. Material balances for the irons showed that the segregation of molybdenum to the phosphide was practically complete. Considering heats 511 and 513, the balances are as follows:

Heat No. 511

Average analysis — 0.75 per cent molybdenum and 0.95 per cent phosphorus

Eutectic analysis — 3.96 per cent molybdenum and 4.37 per cent phosphorus

Basis—1 pound iron

Phosphorus in eutectic = $0.0095 - 0.0012 = 0.0083$ pound
(solubility of phosphorus = 0.12 per cent as shown in Fig. 4)

Weight of eutectic = $0.0083 \times \frac{100}{4.37} = 0.19$ pound

Weight of molybdenum in eutectic = $0.19 \times 0.0396 = 0.00753$ pound

Total molybdenum in 1 pound iron = 0.0075 pound

Heat No. 513

Average analysis — 0.75 per cent molybdenum and 1.60 per cent phosphorus

Eutectic analysis — 2.23 per cent molybdenum and 4.48 per cent phosphorus

Basis—1 pound iron

Phosphorus in eutectic = $0.0160 - 0.0012 = 0.0148$ pound

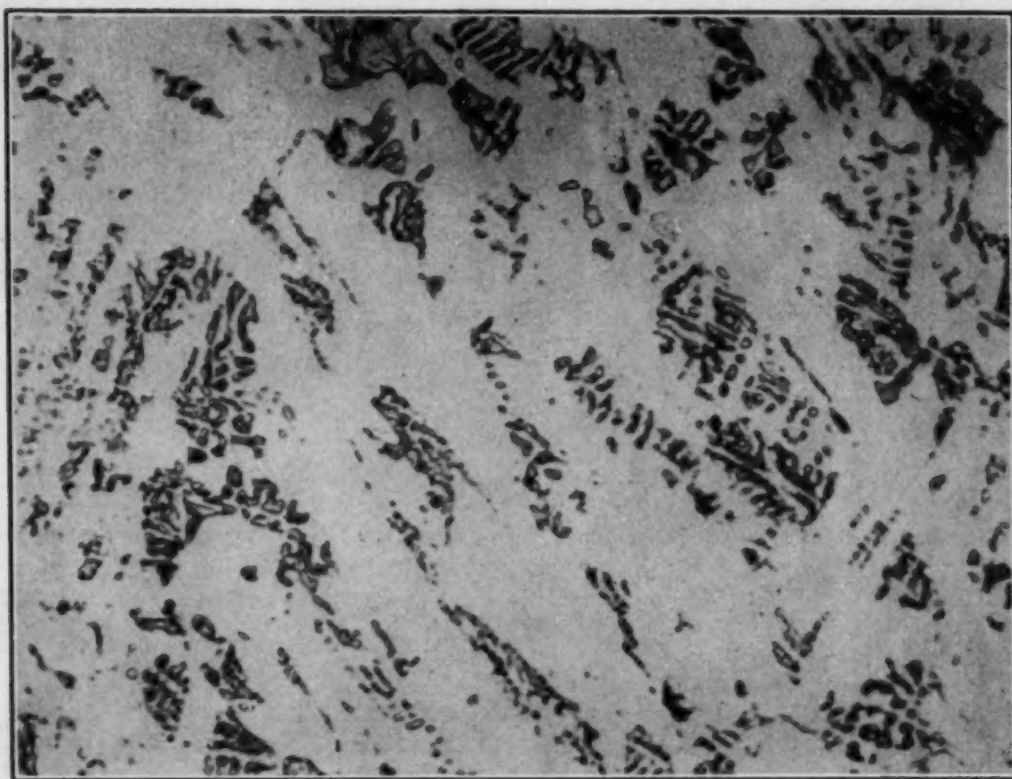


Fig. 7—Microstructure of the Small Pellet of Phosphide Eutectic from Top of the 3-Inch Casting of Heat N6. Compare Structure with the Eutectic Areas in Fig. 5. $\times 1000$.

$$\text{Weight of eutectic} = 0.0148 \times \frac{100}{4.48} = 0.33 \text{ pound}$$

$$\text{Weight of molybdenum in eutectic} = 0.33 \times 0.0223 = 0.00738 \text{ pound}$$

Since castings usually run to much lower phosphorus contents than those discussed above, it was necessary to establish the maximum attainable molybdenum content of the phosphide eutectic, in order to appraise the seriousness of the segregation. For this purpose heat No. 514 with 2.65 T.C., 2.25 Si, 1.20 Mn, 1.75 Ni, 2.50 Mo, and 1.60 P, was made. The high phosphorus content was selected to insure that a sufficient weight of the extruded pellets would be available for analysis. The analysis of the pellets from the 3-inch casting of this heat and the comparative analysis of the ternary eutectic in unalloyed irons are given below:

	Heat No. 514	Unalloyed Irons
Per cent total carbon	2.12	1.96
Per cent silicon	1.15	1.00
Per cent phosphorus	4.47	7.04
Per cent molybdenum	5.85
Per cent nickel	1.65



Fig. 8—Microstructure of the Center of a 3-Inch Casting from Heat 514. Analysis: 2.65 Total Carbon, 2.25 Silicon, 1.20 Manganese, 1.75 Nickel, 2.50 Molybdenum, and 1.60 Phosphorus. Matrix Molybdenum Content is 0.87 Per Cent. Note Pearlite Envelope Surrounding the Eutectic Area. $\times 1000$.

A material balance showed that 0.87 per cent molybdenum remained in the matrix, which indicates that the pellets had achieved the maximum molybdenum content and were probably of quaternary eutectic composition.

These results demonstrate conclusively the reasons for the differences in temperature and "time" of solidification in molybdenum alloyed irons as compared with unalloyed irons. The lower temperature is undoubtedly due to the introduction of a fourth alloy, molybdenum, into the ternary iron-carbon-phosphorus system, while the longer "time" is due to the larger quantity of phosphide eutectic formed in the alloyed irons.

The practical significance of the segregation is apparent upon calculation of the quantity of molybdenum combined in the phosphide in an ordinary gray iron to which a molybdenum alloy addition had been made. In a casting of ordinary foundry phosphorus content, 0.35 to 0.40 per cent, the quantity of molybdenum uselessly combined in the quaternary eutectic before any appreciable quantity can

be released to the matrix is 0.30 to 0.36 per cent. Therefore, small additions of molybdenum to an ordinary gray iron, in which no attempt is made to keep the phosphorus to low levels, are useless.

It might be assumed from the foregoing results that sufficient molybdenum could be added to satisfy the demands of the phosphide eutectic, and the matrix could then be alloyed in the usual manner. However, metallographic examination of the casting from heat No. 514 (the high phosphorus, high molybdenum heat) showed that, although the calculated matrix molybdenum content was 0.87 per cent, and the nickel content was about 1.75 per cent, pearlite was present in envelopes around the phosphide eutectic areas. This structure is shown in Fig. 8.

It is apparent, therefore, that in the attempted production of the highest test gray irons in which the acicular structure is to be obtained by alloying with molybdenum it is essential that the phosphorus content be maintained at a preferable maximum of about 0.12 per cent.

PHOSPHIDE SOLIDIFICATION TEMPERATURES

It will be recalled that the plot of phosphide solidification temperature in the alloyed irons versus phosphorus content shown in Fig. 3 was discontinuous, with a "shelf" indicating no influence of phosphorus on the solidification temperature from low phosphorus contents to about 0.70 per cent, and continuously higher solidification temperatures with increasing phosphorus content above 0.70 per cent. A phosphorus-molybdenum material balance for a 0.75 per cent molybdenum iron shows that the phosphide will be saturated with molybdenum at phosphorus contents up to 0.72 per cent, but that at higher phosphorus contents the availability of molybdenum limits the content in the phosphide to less than saturation, about 5.85 per cent. Therefore, up to 0.70 per cent phosphorus the phosphide in the iron will be of constant composition, but with increasing phosphorus in the iron the molybdenum content in the phosphide will be reduced.

Thus, the "shelf" in the curve of Fig. 3 marks the limits of constant phosphide composition in a 0.75 per cent molybdenum iron. The second section of the curve indicates decreasing molybdenum content in the phosphide, which reflects in the curve as higher solidification temperatures. The curve would, presumably, become asymptotic to the curve for the plain irons shown in the same figure.

THE DIFFUSION OF MOLYBDENUM BETWEEN AUSTENITE AND PHOSPHIDE

It is apparent from an examination of the graphs of Fig. 2 that the nickel-molybdenum alloyed irons solidified with full alloy content in the austenite. The liquidus temperatures of the alloyed irons were consistently lower than in the unalloyed irons at all phosphorus levels, and the eutectic temperatures were consistently higher. However, it is apparent also that the molybdenum had segregated to the phosphide eutectic liquid by the time it was ready to solidify, as evidenced by the lower temperature of solidification and the longer time required in the alloyed irons. The molybdenum must, therefore, have diffused from the solid austenite to the liquid phosphide eutectic during the cooling from the iron-iron carbide eutectic to the iron-carbon-phosphorus-molybdenum eutectic temperatures.

In order to determine whether or not a reversal of the diffusion would occur upon reheating the alloyed irons to above their eutectic melting temperature, a series of remelting experiments was conducted. For this purpose $\frac{3}{4}$ -inch cubic sections were removed from the centers of the castings and reheated to the eutectic temperature and melted.

Holes were drilled into the small cubic sections, and chromel-alumel thermocouples, with beads protected by a thin coating of alundum cement-sodium silicate mixture, were inserted. The assembly was then placed in holes drilled into refractory bricks, covered with a slurry of alundum cement and water, and baked at 500 degrees Fahr. (260 degrees Cent.) after air drying. This arrangement was selected in order that the heating rate during the remelting procedure would be nearly the same in the equipment available as the cooling rate of the original castings.

The brick was placed in a Hayes Globar element electric furnace and heated, under constant power input, to 2200 degrees Fahr. (1205 degrees Cent.). Temperature readings were made each 10 seconds during the heating from 1000 degrees Fahr. (540 degrees Cent.). A plot of temperature versus time gave a characteristic heating curve with "halts" at the austenite transformation temperature, phosphide eutectic melting temperature, and iron-carbon eutectic temperature. In all, six sections were remelted, and heating curves were obtained for each. The data taken from these curves are presented in Table V and plotted in Figs. 9 and 10.

As shown in Fig. 9, increasing phosphorus content from 0 to 0.60 per cent raised the austenite transformation temperature 40 to 50 degrees Fahr., but with further increases above 0.60 per cent the transformation temperature was lowered. The initial increase in temperature was probably caused by the introduction of phosphorus in the iron lattice. After the maximum solubility of phosphorus

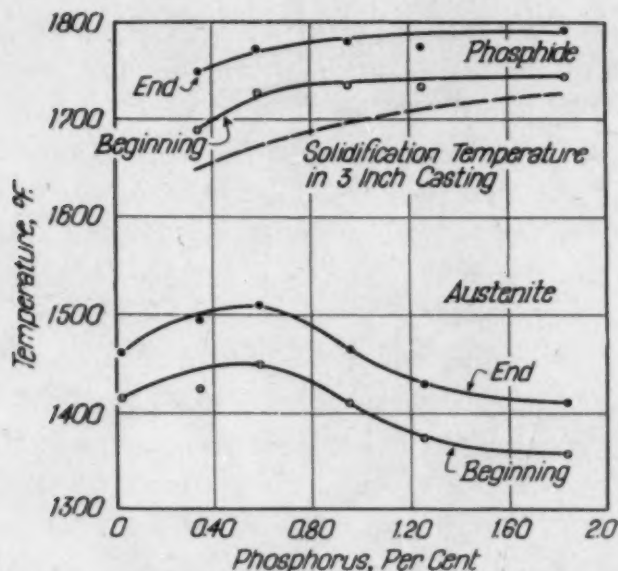


Fig. 9—Influence of Phosphorus on the Austenite Transformation Temperature Range and Phosphide Melting Range on Reheating Sections of 3-Inch Castings. (Base Analysis—2.65 Total Carbon, 2.25 Silicon, 1.75 Nickel, 0.75 Molybdenum.)

had been exceeded no further significant effect with increasing phosphorus was noted until the content became sufficiently high to permit the removal of a significant quantity of molybdenum from the matrix to effect a lowering of the austenite transformation temperature. This quantity was apparently reached at about 0.60 per cent phosphorus. The temperature then decreased and approached an asymp-

Table V
Transformation Temperatures on Reheating Sections from 3-Inch Cylinders

Heat No.	Per Cent P	Temperature (°F.)					
		Austenite		Phosphide		Eutectic	
		Beg.	End	Beg.	End	Beg.	End
1	0.016	1415	1462	None	None	2093	2118
8	0.34	1424	1494	1690	1749	2064	2105
5	0.58	1450	1511	1727	1772	2040	2097
7	0.95	1410	1465	1735	1780	2000	2065
2	1.25	1375	1430	1732	1775	1967	2025
10	1.83	1357	1410	1745	1792	1926	1983

tote as the molybdenum content of the matrix approached zero.

The phosphide melting range was slightly higher on reheating than on cooling the original casting. This is a commonplace behavior.

As shown in Fig. 10, the iron-iron carbide eutectic melting temperatures were influenced in a very unusual manner by increasing phosphorus content. At lower phosphorus contents the eutectic temperatures were somewhat higher than in the original castings, which

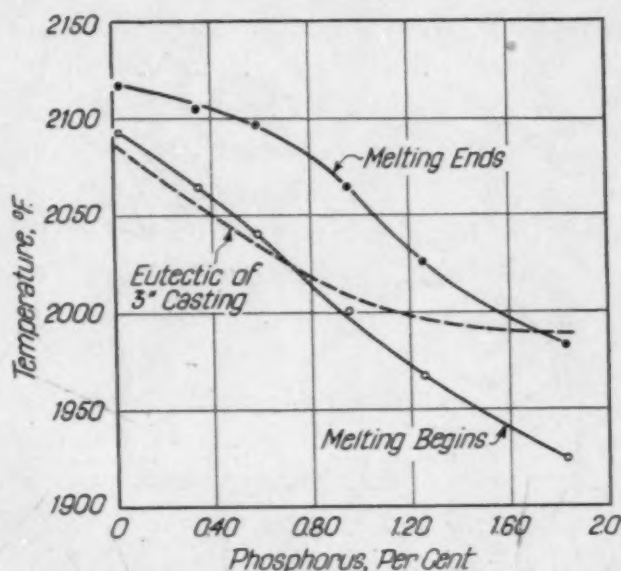


Fig. 10—Influence of Phosphorus on the Eutectic Melting Temperature Range on Reheating Sections from 3-Inch Castings. (Base Analysis—2.63 Total Carbon, 2.25 Silicon, 1.75 Nickel, 0.75 Molybdenum.)

would have been expected. However, at phosphorus contents above about 0.70 per cent the remelting of the eutectic began on temperatures lower than the eutectic temperature on cooling. In fact, the eutectic of the 1.83 per cent phosphorus iron was completely remelted by the time its temperature on reheating had reached the eutectic temperature on cooling.

These results answer the question originally raised as to whether or not a reversal of the rapid diffusion of molybdenum from austenite to phosphide on cooling would be realized on reheating. It is obvious that this is not the case. Since, as shown in Fig. 2, unalloyed irons had lower eutectic temperatures than alloyed irons, it follows that, in reality, the temperature measured on reheating the cubic sections was the temperature of melting of an unalloyed eutectic,

in other words, one devoid of molybdenum. The nickel content of the matrix of all the alloyed irons was about 1.75 per cent as indicated before; however, since it is known that nickel has little effect on the eutectic temperature, it can be ignored in this case.

Photomicrographs at $\times 100$ and $\times 500$ of the remelted section from heat No. 7 (0.95 per cent phosphorus) are shown in Fig. 11. The specimen was heated to 2200 degrees Fahr. (1205 degrees Cent.), held about 5 minutes, then removed from the furnace and allowed to cool in the air. The refractory brick crucible reduced the cooling rate somewhat, although the rate was considerably greater than in the original 3-inch casting. This is demonstrated by the presence of eutectiform (undercooled) graphite in the air-cooled structure.

The areas which had been melted on reheating can be readily distinguished from those areas not remelted by their martensitic structure. Apparently the rapid cooling rate between the iron-iron carbide and iron-phosphorus-carbon eutectic temperature inhibited the segregation of molybdenum which, in combination with the rapid cooling rate at subeutectoid temperatures, permitted the remelted austenite to transform to martensite. As a matter of fact, it is possible that the actual molybdenum content of the martensitic areas was greater than the average 0.75 per cent in the casting itself. Since in the original casting all the molybdenum was combined with the phosphide eutectic, all of which melted on reheating, the molybdenum content of the liquid present at 2200 degrees Fahr. (1205 degrees Cent.) was considerably greater than 0.75 per cent (only a fraction of the proeutectic austenite melted). Therefore, if the subsequent cooling rate were rapid enough, the austenite could retain more than 0.75 per cent molybdenum.

FREEZING AND REMELTING OF ALLOYED GRAY IRON CASTINGS

On the basis of the preceding observations, the solidification and remelting of nickel-molybdenum-phosphorus alloyed gray iron castings may be described in the following manner: At temperatures above the liquidus the alloys are distributed homogeneously in the entire melt. On cooling to the liquidus temperature primary austenite of apparently full alloy contents, that is, nickel and molybdenum, with probably about 0.12 per cent phosphorus, begins to separate from the melt. This continues to the eutectic temperature. The eutectic reaction consists in the separation of saturated austenite

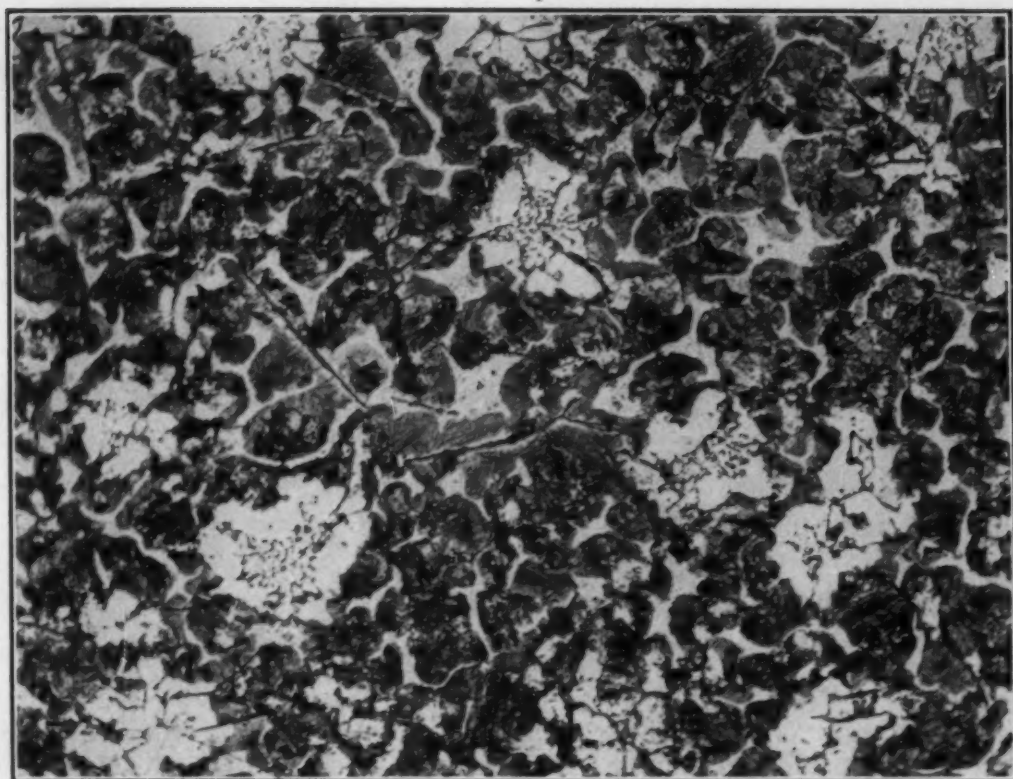


Fig. 11—Microstructure of a Section from the 3-Inch Casting of Heat No. 7 (2.65 Total Carbon, 2.25 Silicon, 1.20 Manganese, 1.75 Nickel, 0.75 Molybdenum, and 0.95 Phosphorus). After Partial Remelting at 2200 Degrees Fahr. Followed by Retarded Cooling in Air. Note the Adjacent Pearlite and Martensite Grains, and the Eutectic Form Graphite in the Upper Photograph. Upper— $\times 100$. Lower— $\times 500$.

and graphite or carbide from the eutectic liquid. During this entire solidification, the phosphorus, except for roughly 0.12 per cent, remains in the liquid state with ternary eutectic composition.

On further cooling, the liquid ternary eutectic receives molybdenum from the matrix until the quaternary eutectic composition is achieved or until nearly all the molybdenum has been removed from the austenite. During this chemical change, however, other reactions, which result in the extrusion of the small pellets previously described, occur. First, the quantity of eutectic is increased as evidenced by the low phosphorus content in the eutectic obtained in the alloyed irons. Second, the surface of the casting cools and contracts, thus exerting a compressive force on the interior of the casting. At high phosphorus contents the combination of expansion and contraction is sufficient to cause actual rupture of the casting surface and the formation of the small pellets.

On reheating, any phosphide eutectic present melts first, and the molybdenum combined therein is not available for alloying the austenite at the iron-iron carbide eutectic. At low phosphorus levels the quantity of molybdenum tied up in the phosphide is not great, so the iron-iron carbide eutectic temperature is at temperature slightly above the original solidification temperatures. As the phosphorus content is increased to higher levels, however, the quantity of molybdenum in the phosphide becomes greater, and the austenite becomes unalloyed insofar as molybdenum content is concerned. Thus, it is melted at a lower temperature which corresponds to the temperature for an unalloyed iron (refer to Fig. 2).

SUMMARY AND CONCLUSION

It has been shown in the present paper that molybdenum forms a complex quaternary eutectic with phosphorus, iron, and carbon in alloyed gray cast iron. The composition is such that for each unit of phosphorus above that amount soluble in iron, 0.12 per cent, 1.3 units of molybdenum are removed from the matrix.

The chemical composition of the complex eutectic obtained in the alloyed iron was:

	Per Cent
Carbon	2.10
Silicon	1.10
Nickel	1.65
Phosphorus	4.40
Molybdenum	5.85
(Balance Iron)	

In low phosphorus, molybdenum alloyed irons the eutectic composition probably approximates the above; however, unless the molybdenum content exceeds 1.3 (per cent phosphorus—0.12), practically all the molybdenum is present in the phosphide, and the molybdenum content of the phosphide is limited by the availability.

As a consequence of the molybdenum segregation, a pearlitic structure may be obtained in a molybdenum alloyed iron, although the average alloy content yields an acicular type structure.

The diffusion of molybdenum to the phosphide eutectic takes place between solid austenite and liquid phosphide at between their respective solidification temperatures. On reheating, however, the reverse reaction does not occur. On remelting an alloyed iron with high phosphorus content it was found that the eutectic melting temperature approached the temperature of unalloyed irons due to the failure of the molybdenum to rediffuse to the solid austenite.

From a practical standpoint, the addition of small quantities of molybdenum to an ordinary gray iron in which no attempt is made to hold the phosphorus content to low levels is useless due to the practically complete segregation of molybdenum to the phosphide eutectic.

ACKNOWLEDGMENT

The writers are indebted to the members of the staff of the Department of Chemical and Metallurgical Engineering of the University of Michigan for helpful suggestions and guidance during the investigation. Thanks are extended to the Department of Engineering Research for financial assistance in obtaining certain equipment and data.

Special thanks are due the undergraduate and graduate students who so kindly assisted in obtaining the data.

DISCUSSION

Written Discussion: By V. A. Crosby, Climax Molybdenum Company of Michigan, Detroit.

The authors' findings add substantially to our fund of knowledge on the subject of alloy phosphide eutectics. The new information, regarding these eutectic phosphides which the authors have given us, will serve as a basis for future investigations. The authors deserve credit for a wise choice of analyses for this investigation. The selection of a critical composition for a section size which will just produce a bainitic structure in 3-inch sections when poured at

2650 degrees Fahr. (1455 degrees Cent.) is evidence that a great deal of study preceded the authors' experiments. The information contained in this paper offers, for the first time, a technical explanation for a phenomenon which a number of foundrymen and metallurgists have observed, but have never been able to explain.

The authors' experiments established the following facts under the conditions of their investigation: A—That the effectiveness of a molybdenum addition on austenite transformation decreases in the presence of increasing percentages of phosphorus. B—That the phosphide eutectic in molybdenum alloyed irons is molybdenum-rich. C—That molybdenum depresses the liquidus temperature and raises the eutectic (iron-iron carbide) temperature. These

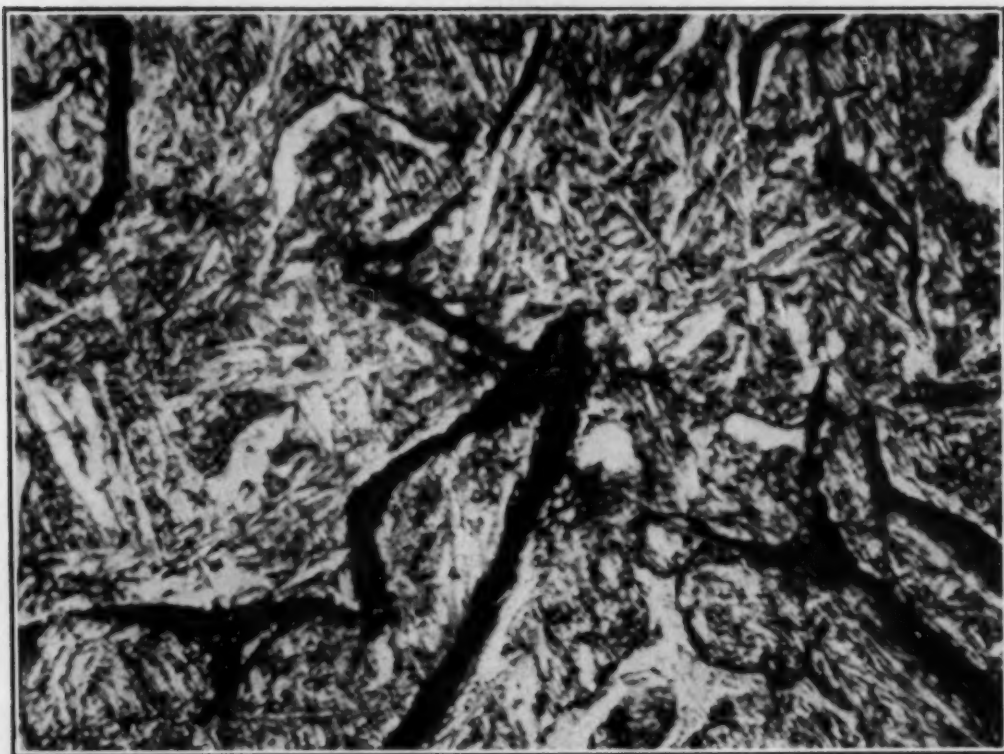


Fig. A

findings may well have practical significance in sections in which cooling rates are slow and near-equilibrium conditions prevail. However, information to follow does not confirm the authors' findings when referring to castings of thin sections and in sections represented by 1.2-inch diameter test bars.

The writer feels that the authors have been a little too comprehensive in their conclusions as stated in the last paragraph of the paper: "From a practical standpoint, the addition of small quantities of molybdenum to an ordinary gray iron in which no attempt is made to hold the phosphorus content to low levels is useless due to the practically complete segregation of molybdenum to the phosphide eutectic". No exception is taken to the quoted statement if the sections are those represented by the 3-inch casting used for the data in this

paper. However, no mention is made of this point; and it cannot be disregarded when segregation is the subject under consideration. Data, accumulated through experience with plain and alloyed gray irons containing up to 0.55 per cent phosphorus in sections from $\frac{1}{8}$ inch to 1.2 inch, furnish results contradictory to those predicted from the authors' formula "... unless the molybdenum content exceeds 1.3 (per cent phosphorus —0.12) practically all the molybdenum is present in the phosphide. . . .". Sections represented by 1.2-inch diameter test bars (and this is the yardstick for measuring the quality of nearly all alloyed iron), and smaller, cool far too fast to permit the degree of segregation indicated by the authors in their study of the 3-inch section test bars.

Fig. A shows an acicular structure obtained by the use of 0.43 per cent molybdenum in a 0.35 per cent phosphorus-copper iron:

Analysis	Per Cent
Total Carbon	3.57
Combined Carbon	0.62
Graphitic Carbon	2.95
Manganese	0.55
Silicon	3.07
Sulphur	0.06
Phosphorus	0.35
Molybdenum	0.43
Copper	0.85

By calculation from the authors' formula, only 0.13 per cent molybdenum could be present to effect the retardation of austenite to bainite as shown in the micrograph.

Table A shows data compiled from 1.2-inch diameter test bars made in a production foundry from cupola metal containing 0.50 to 0.55 per cent phosphorus:

Table A

Analysis:	Bar LF-1 Per Cent	Bar LF-2 Per Cent
Total Carbon	3.48	3.44
Combined Carbon	0.72	0.78
Graphite	2.76	2.66
Manganese	0.62	0.61
Silicon	2.17	2.16
Sulphur	0.122	0.110
Phosphorus	0.523	0.540
Molybdenum	0.037	0.62
Nickel	Trace	0.12
Chromium	0.12	0.12
Physical Properties:		
Transverse Bars	LF-1	LF-2
	Pounds	Pounds
Transverse Load (18-inch Centers)	2376	2640
Deflection	0.168	0.22
B.H.N.	228	241
Tensile Bars	LF-1	LF-2
Tensile strength, psi.	29,920	36,960
B.H.N.	217	241

The increase in tensile strength of 7040 psi., with the use of 0.62 per cent molybdenum, is within the range of normal expectancy. On the basis of calculation proposed by Rote and Wood, however, no molybdenum would be available to bring about this increase of strength.

In Table B, laboratory data are presented from results obtained in testing 1.2-inch diameter bars produced from an induction heat of gray cast iron containing 0.35 per cent phosphorus:

Table B

1. Chemical Analysis of Arbitration Test Bar Identification	Bars 1.2-Inch Diameter			
	1	2	3	4
	Per Cent			
Total Carbon	3.30	3.32	3.28	3.25
Combined Carbon	0.82
Graphite	2.48
Manganese	0.62
Silicon	1.81
Sulphur	0.085
Phosphorus	0.350
Molybdenum	0.008	0.26	0.54	0.88
Nickel	None
Chromium	None
2. Brinell Hardness				
As cast	217	228	241	255
3. Transverse Tests				
Load, pounds	2,415	2,660	2,925	3,270
Deflection, inches	0.255	0.272	0.269	0.277
4. Tensile Strength, psi.	40,500	45,800	49,800	52,400
5. Impact Strength, foot/pounds	35.4	35.7	44.0	47.6

As a basis for comparison using No. 1 and No. 2 bars, an increase of 5000 psi. is observed with 0.26 per cent molybdenum. Here again, on the basis of the authors' (Rote and Wood) calculation, there would be no molybdenum available.

Table C shows data compiled from 1.2-inch diameter test bars made in a production foundry using cupola metal with the alloys added to the ladle:

Table C

Analysis:		331-B	331-A Per Cent	331-P Per Cent
Bar Numbers				
Total Carbon	3.34
Combined Carbon	0.74
Graphite	2.60
Manganese	0.55
Silicon	1.46
Sulphur	0.158
Phosphorus	0.377
Molybdenum	None	0.32	0.41
Nickel	0.05
Chromium	None
Physical Properties:				
Transverse (18-inch Centers)	2,227 Pounds	2,340 Pounds	2,525 Pounds	
Deflection	0.286 Inch	0.261 Inch	0.300 Inch	
Brinell	207	217	223	
Tensile	35,600 Pounds	41,800 Pounds	42,240 Pounds	
Brinell	202	217	217	

The results are in close agreement with data shown in Table B. The alloy addition of 0.32 per cent molybdenum to a base iron containing 0.377 per cent phosphorus increased the tensile strength 6200 psi.—a figure well within our normal expectancy for 0.32 per cent molybdenum. The authors' calculations would indicate a mere 0.02 per cent molybdenum available for this job.

In Table D, data are shown comparing a 0.42 per cent phosphorus iron unalloyed and alloyed with 0.27 per cent molybdenum. It would be evident from Rote and Wood's calculations that there would be insufficient molybdenum to take care of the phosphorus by 0.12 per cent, yet we observe an increase in tensile strength and an increase in hardness somewhat above the normal expectancy.

Table D

Chemical Analysis:		
	Plain Iron Per Cent	Molybdenum Iron Per Cent
Total Carbon	3.45	3.41
Combined Carbon	0.59	0.78
Graphite	2.86	2.63
Manganese	0.50	0.48
Silicon	2.20	2.10
Sulphur	0.062	0.058
Phosphorus	0.45	0.42
Molybdenum	None	0.27
Nickel	None	None
Chromium	Trace	Trace
Physical Properties:		
	Tensile Strength Psi.	Brinell
Plain Iron	30,800	202
Molybdenum Iron	38,280	228

In calling attention to the value of molybdenum, even in irons containing up to 0.55 per cent phosphorus (and in sections represented by 1.2-inch diameter test bars or less), the writer does not wish to leave the impression that higher phosphorus alloyed irons than those used commercially could be considered quite satisfactory. Low phosphorus content is desired for other reasons than for strength. The prevailing practice of maintaining phosphorus at 0.15 per cent maximum for nearly all alloyed irons has merit and is the result of many years of experience. In some designs and for some particular applications, alloyed irons with phosphorus content reduced to 0.05 per cent are required to prevent segregation.

Written Discussion: By J. S. Vanick, metallurgist, International Nickel Co., Inc., New York.

The authors have made an important contribution to our knowledge of cast irons. I believe its importance ranges beyond the confines of its title and provides us with some markers on the map of compositions within which a safe course may be steered toward producing high strength cast irons.

The problem of whether you get an acicular structure or not might be comparable in importance to the similar foundry problem of whether you get an unmachinable white iron casting or a machinable gray one. In both cases, the speed of crystallization or segregation is extremely rapid and control is commonly exercised by adjusting the composition rather than attempting to do anything with the cooling rate.

Information that an appreciable segregation occurs in low phosphorus castings has been known to some foundrymen. Roll makers, for example, have examined the "sweat" shown in Fig. 6 that occurs in larger particles on the

necks of rolls. In a case of this kind, our laboratory obtained the following results. The segregation of phosphorus and molybdenum is striking.

Composition	Roll	"Sweat"	Change from Roll Composition
C	2.90	3.11	+ 7
Si	0.55	0.43	- 22
Mn	0.22	0.11	- 50
Mo	0.12	0.53	+ 340
S	0.08	0.11	+ 37
P	0.45	2.50	+ 450

I have long felt the need to specify the composition of iron that is desired for a specific purpose because physical tests of the usual type are inadequate and may not define the character of the product in its final desired form. I have observed specifications for nickel-molybdenum high strength irons which permitted up to 0.60 per cent phosphorus for castings that are to be subsequently heat treated. The difficulties invited by the failure for the structure of these irons to become acicular either "as cast" or alternately, if successful, "as cast", to lose this quality upon being heat treated, poses a serious problem for either the producer of the castings or the processor of them in arriving at the desired quality in the finished articles.

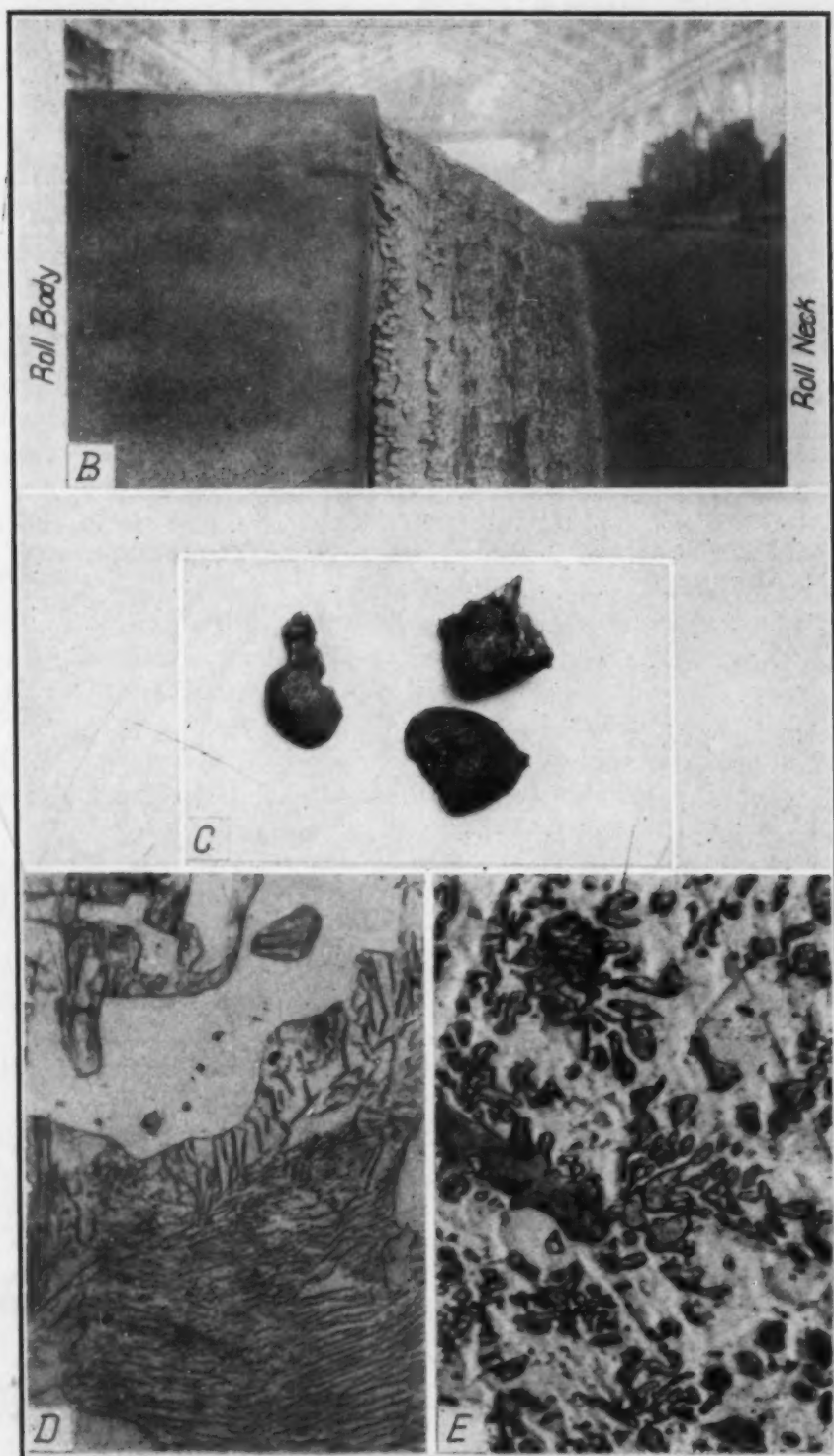
Many of us build our ideas of the behavior of cast iron upon the basic concept that gray cast iron can be considered to be a steel interspersed with graphite. We are aware that the silicon and carbon contents of cast iron substantially affect the relationship between it and steel. We usually fail to exercise the same degree of respect that the steel maker exercises regarding the presence of other elements and the risks of contamination and segregation that may occur. Consequently, the "S" curves or "TTT" curves for a 4640 type of nickel-molybdenum steel, for example, which might be instructive for a low-phosphorus cast iron, would be practically useless in predicting the behavior of high-phosphorus, nickel-molybdenum cast iron whose structure finished pearlitic when it should have been acicular. We are all aware, I believe, that for two irons of the same strength, the acicular structured one will be tougher and possess a greater resistance to impact and fatigue. The authors might have included some measure of this advantage to emphasize the practical importance of their work.

While this work should be helpful in guiding us toward the achievement of the acicular structure where this objective is desired, it suggests extending the study to include:

- (a) the rate at which the segregate forms
- (b) some measure of the rate and degree of segregation for changes in carbon content
- (c) the stability or instability of the segregate at different temperature levels.

Written Discussion: By George M. Biggert, laboratory chemist, Canton Research Department, United Engineering and Foundry Co., Canton, Ohio.

In our study of the paper, "Segregation of Molybdenum in Phosphorus-Bearing Alloyed Gray Cast Iron" by Rote and Wood, we have found the confirmation of several beliefs which we had formed at the time we worked on this subject.



Figs. B to D—Occurrence of Roll Bleeds and Their Microstructures. B—Appearance on Roll. C—After Removal from Roll. D—Bleed Microstructure. $\times 500$. E—Bleed Microstructure. $\times 50$.

One of our patented U. E. & F. Co. chill grain roll analyses is:

C	Si	S	Mn	P	Ni	Cr	Mo
3.35	0.95	0.065	0.35 to 0.40	under 0.100	0.15	0.80	0.30

If this phosphorus content were allowed to climb to 0.20 or 0.25 per cent, by early experience we know the roll would give poor service. This would mean, as Rote and Wood have so capably shown, that only 0.10 to 0.20 per cent of molybdenum would be in the matrix and we feel reasonably certain that this loss of otherwise effective molybdenum is responsible.

Our Canton United Plant some years ago found that chemical laboratory tests cut from the roll fillets gave us a satisfactory average roll analysis. This is true because the fillet gives the best representation of an average area between the chill and gray irons found in the roll. The minute the phosphorus content reached a point where segregation of the phosphide eutectic was possible we had an analytical puzzle in trying to correlate our phosphorus and molybdenum results with the calculated results. This undoubtedly was a trouble created through segregation.

Had Rote and Wood cast their 3 by 4½-inch cylinders in a chill mold as we cast rolls instead of in a core mold, I believe they would have found the bleeding to be far more extensive. In the case of rolls this is true, as can be shown by Fig. B. This picture of a 34 by 126-inch Plate Mill Roll shows the bleeding to extend from under the chill of the body all the way along the taper to the neck diameter. Fig. B illustrates an extreme case of bleeding due to segregation. The paper just read shows that this bleeding was taking place from the time the iron-iron carbide eutectic temperature was reached until the quaternary phosphide eutectic had frozen. This means that the temperature range for this reaction is from approximately 2000 to 1725 degrees Fahr. (1095 to 940 degrees Cent.), which in this large 34 by 126-inch roll would require a time interval of an hour and one-half to two hours. A lot of bleeding can and did take place.

In Fig. C we have the removed bleeds showing the root or feeding stems and the effect of the restriction where they were squeezed as these bleeds were formed.

Fig. D of the bleeds at $\times 500$ shows the phosphide eutectic, pearlite, and free ferrite to be present.

Fig. E at $\times 50$ shows the general arrangement in the bleed microstructure.

In Fig. F is plotted, in comparison, the analysis of the roll and the analysis of the roll bleeds as found at the cope neck of three different rolls. The elements carbon, silicon, manganese and nickel in the bleeds were found in lower percentages than the percentages of the corresponding elements in the average roll analysis. These elements are noted as being graphitizers. The sulphur, phosphorus, molybdenum and chromium show the reverse phenomena in that the bleeds have the higher percentages for the exception of the sulphur in one roll, S-1035. These elements are noted as being the carbide stabilizers. It is observed that the phosphorus and molybdenum in the bleeds rose as much as 1100 and 750 per cent, respectively, above these same elements in the average roll analysis.

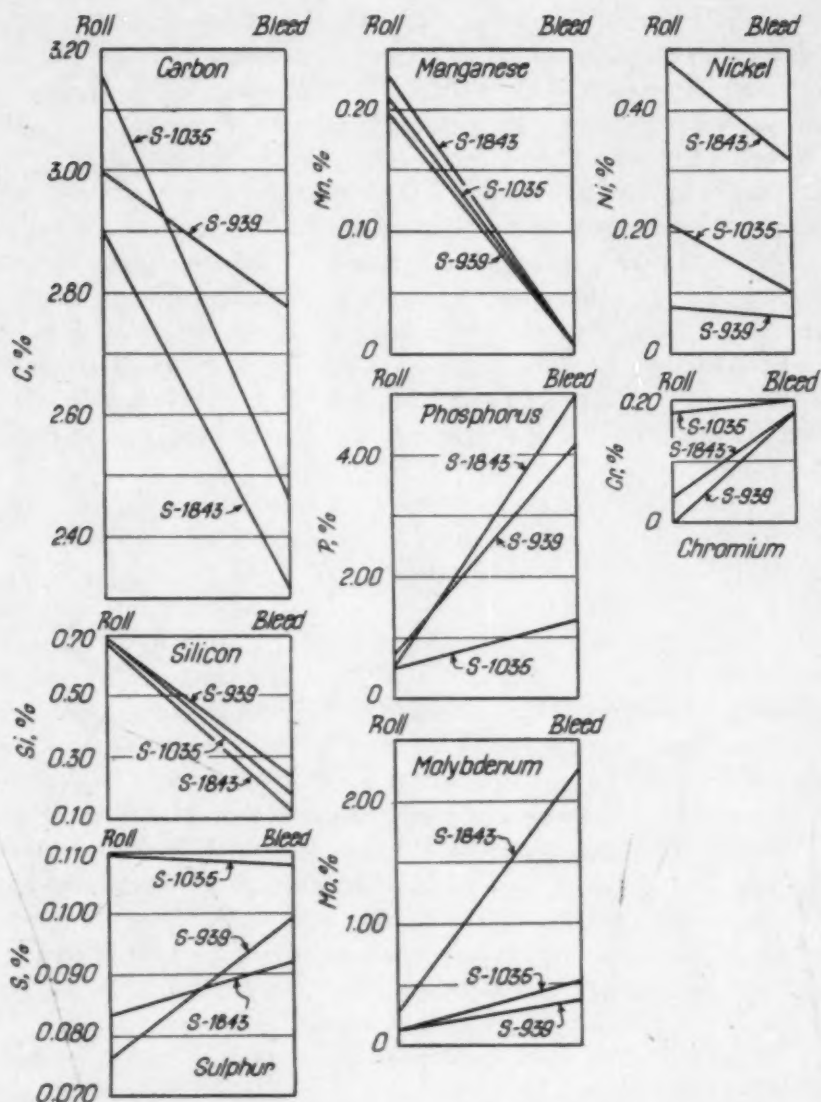


Fig. F—Roll Analyses Versus Cope Bleed Analyses on Rolls S-939, S-1035, S-1843.

From this viewpoint, any casting where chills must be applied, such as might be done in trying to accelerate the cooling of a wheel hub, this segregation and consequent bleeding may become of very great importance, when one is trying to obtain a certain analysis and the consequent mechanical properties.

I want to take this opportunity to praise and congratulate both Mr. Rote and Mr. Wood on the exceptionally neat work which they have done in the preparation of this paper.

Authors' Reply

The interest exhibited by the discussers in the preparation of written contributions and the constructive comments which they have presented are appreciated by the authors. Considerable data of importance were presented.

Mr. Crosby's discussion shows conclusively the beneficial effects of rather small additions of molybdenum in light section castings of relatively high phosphorus content. However, it shows also the inadequacy of the 1.2-inch test bar as a yardstick for measuring the quality of alloyed irons for any casting more than $\frac{1}{2}$ to $\frac{3}{4}$ inch thick. Calculations show that the ratio of area to volume in a 1.2-inch diameter infinite cylinder is 3.33 square inches per cubic inch. The same ratio is obtained in an infinite flat plate 0.60 inch thick. It would seem, therefore, that a 1.2-inch arbitration bar and a 0.60-inch plate would cool at the same rate, providing the speed at which heat can be removed from the surface is the controlling factor in the cooling of a casting. Experimental work by the authors showed that the structure, hardness, and tensile strength of 1.2-inch test bars and of plates 0.50 and 0.75 inch thick were similar in a number of different alloyed irons, which indicated that the cooling rates were approximately the same.

Similar area to volume ratio calculations show that a 3-inch diameter infinite cylinder would be comparable to a plate only 1.5 inches thick. Thus, as shown by Mr. Crosby's data, molybdenum additions to relatively high phosphorus irons may be beneficial in a $\frac{1}{2}$ to $\frac{3}{4}$ -inch section, while as shown by the authors' work, they may be totally lost by segregation in a 1.5-inch section. Therefore, if any acceptance standard is to be established for alloyed irons, its section should be based on the predominating critical section in the casting.

The authors acknowledge the profound effect of cooling rate on the degree of segregation in a phosphorus and molybdenum-bearing iron, and suggest this as a good reason for reappraising the arbitration bar standards now in use.

Mr. Vanick's data correlate with those presented by the authors. His comments regarding the problems in heat treating phosphorus-bearing alloyed irons are well taken, since as shown in the paper, re-solution of the molybdenum taken up by the phosphide eutectic did not occur even on heating to above the iron-iron carbide eutectic temperature range. Thus, as he pointed out, "S" curves or "TTT" curves might be misleading.

While no quantitative data were given to show the rate of formation of the complex phosphorus-molybdenum-rich eutectic or its rate of re-solution, it was shown that the segregation occurred in the time interval between the iron-iron carbide and iron-iron phosphide eutectic, about 13 minutes in the 3-inch casting. Further, practically no re-solution of molybdenum occurred until the iron had been reheated to above the iron-iron carbide eutectic temperature. There may be partial answers to his suggested extensions of the investigation.

It is gratifying to learn of the experience reported by Mr. Biggert, since it presents practical evidence of the loss in mechanical properties or roll life when the action of valuable molybdenum was lost by segregation to the phosphide eutectic. Mr. Biggert's plots showing the segregation of the carbide stabilizers in the phosphide and of graphitizers in the matrix of a casting are valuable additions to the data presented in the paper.

Acknowledgment is made of private discussions of the paper by others who were unable to present oral discussions because of lack of time during the technical session.

A RELATIONSHIP BETWEEN HARDENABILITY AND TENSILE STRENGTH OF NORMALIZED STEELS

BY LOUIS A. CARAPELLA

Abstract

The hardenability of a steel has been found, as a general rule, to affect the ratio between the water-quenched hardness and the hardness resulting from any definite rate of cooling according to the equation

$$\frac{H'}{H} = A \log_e D_1 + B.$$

By augmenting this equation through the use of other derived empirical expressions, a relationship between hardenability and tensile strength of normalized steels has been established.

This finding may be mathematically stated as follows:

$$\text{T.S.} = \frac{93,000 \log_e \text{Per Cent Carbon} + 362,700}{-0.85 \log_e D_1 + 3.25} + 5000.$$

A simple chart has been prepared by which the above equation can be readily solved.

The validity of the foregoing relationship has been confirmed in a wide variety of commercially normalized steels. This information will assist in developing new steel compositions with desired combinations of hardenability and normalized tensile properties through an efficient and economical use of alloying elements.

THAT the hardness and tensile properties of steels are related has long been common knowledge to the physical metallurgist. Of similar recognition is the fact that the hardness of steels is more or less governed by the hardenability, depending, of course, on the rate of cooling from the austenitizing temperature. On the basis of these existing evidences, a relationship between hardenability and tensile properties of steel is suggested.

The underlying reason in support of this suggestion is the fact that the inherent factors of hardenability, i.e., chemical composition and grain size, fundamentally influence the eutectoid compositions, transformation temperatures, and rates of reaction, and thereby

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alter the general character of the aggregate structures of steels. Gensamer and his co-workers (1)¹, (2) have quantitatively demonstrated the dependence of tensile properties of steels on the nature of their microstructures.

For commercial interests, a relationship between hardenability and tensile strength of normalized steels has been established. This information, which is useful in that it supplements other predictable properties of steels, is computed from only hardenability values as proposed by Grossmann (3) and the derived equation. Moreover, this relationship augments the possibility of selecting new steel compositions with an efficient and economical use of alloying elements to obtain desired combinations of hardenability and normalized tensile strength.

DERIVATION OF THE RELATION OF HARDENABILITY TO NORMALIZED TENSILE STRENGTH

In the development of the equation correlating hardenability and normalized tensile strength, three independent relationships have been used, namely:

- (a) Hardenability as a function of the ratio of hardness in both the water-quenched and normalized conditions;
- (b) Water-quenched hardness as a function of carbon content; and,
- (c) Hardness as a function of tensile strength.

Before arriving at the final equation, it was necessary to derive within certain limits suitable mathematical expressions for each of these functions. The limits were selected so as to include conditions likely to be experienced in commercial practices. Considerations of these functions will be individually discussed.

Hardenability as a Function of Hardness—Notwithstanding the fact that much work has been done on both the hardness and hardenability of steels, the work of Field (4) is of noteworthy importance because it shows a quantitative correlation between the ideal critical diameter (hardenability) and the ratio of initial-end-hardness to the distance-hardness of a Jominy test bar (see Fig. 1). A further consideration of this work reveals that the general function given at right best defines the relation of hardenability to the hardness ratio:

¹The figures appearing in parentheses pertain to the references appended to this paper.

$$\frac{H'}{H} = A \log_e D_1 + B, \quad (1)$$

where

H' = initial hardness,
 H = distance hardness,
 D_1 = ideal critical diameter, and
 A, B = constants.

Moreover, the function is valid over a wide range of cooling rates, as seen in Fig. 2. As a general case, H' may be assumed as the water-quenched hardness and H as the hardness which results from a particular rate of cooling from the austenitizing temperature.

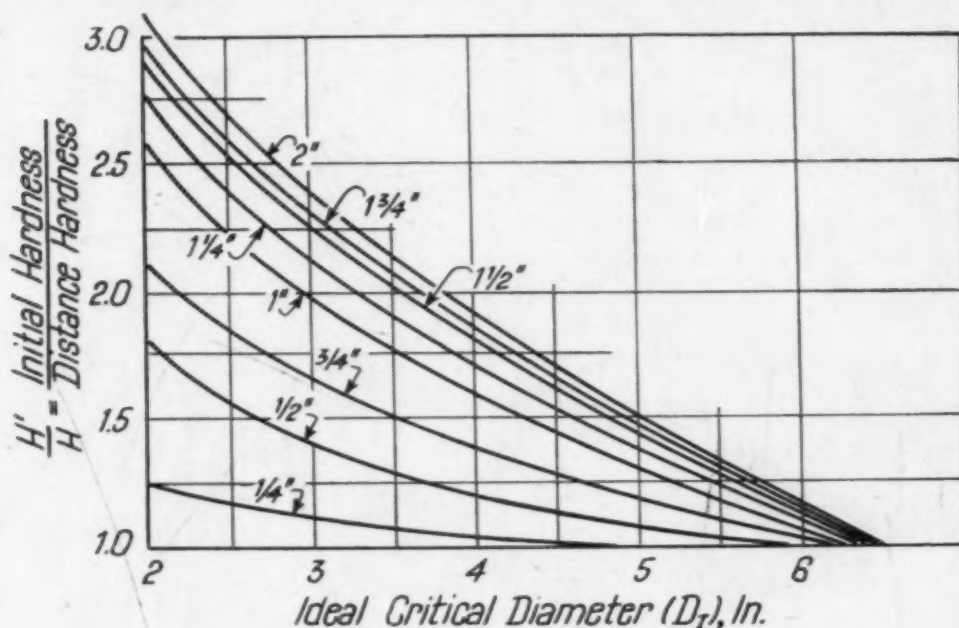


Fig. 1—A Relation Between Hardness Ratio and Hardenability at Various Distances Along a Jominy Test Bar. [According to J. Field (4)].

Water-Quenched Hardness as a Function of the Carbon Content

—For plain carbon and common alloy steels, Burns, Moore, and Archer (5) have demonstrated that the maximum hardness attainable upon quenching depends on the carbon content alone and is independent of the austenitic grain size. From general experiences, however, the hardness of water-quenched steels has been found to fall within the shaded area given in Fig. 3. Between the limits shown the following equation gives the most probable value of hardness:

$$H' = 200 \log_e \text{Per Cent Carbon} + 780, \quad (2)$$

where

H' = water-quenched hardness (BHN).

This equation is in close agreement with the observations of Welchner, Rowland, and Ubben (6).

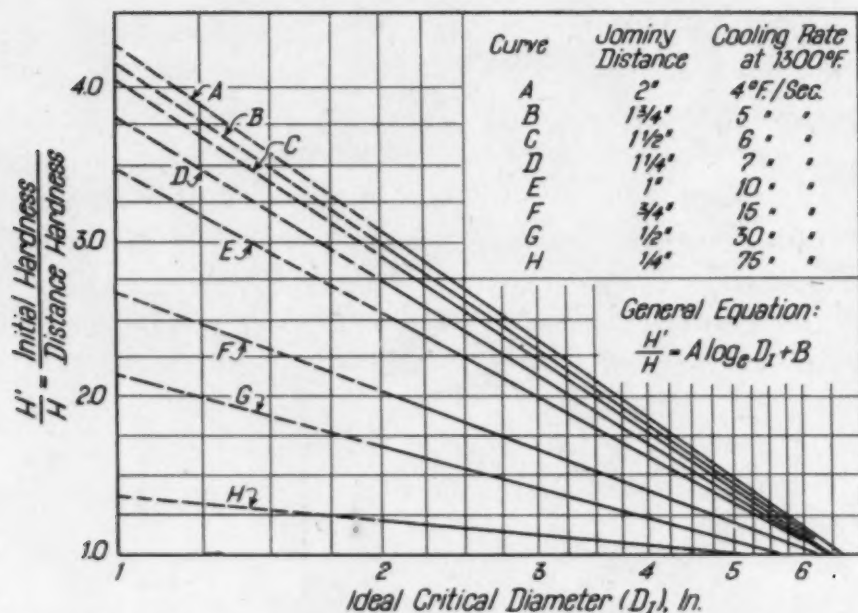


Fig. 2—A General Equation Relating Hardness Ratio and Hardenability for Various Rates of Cooling.

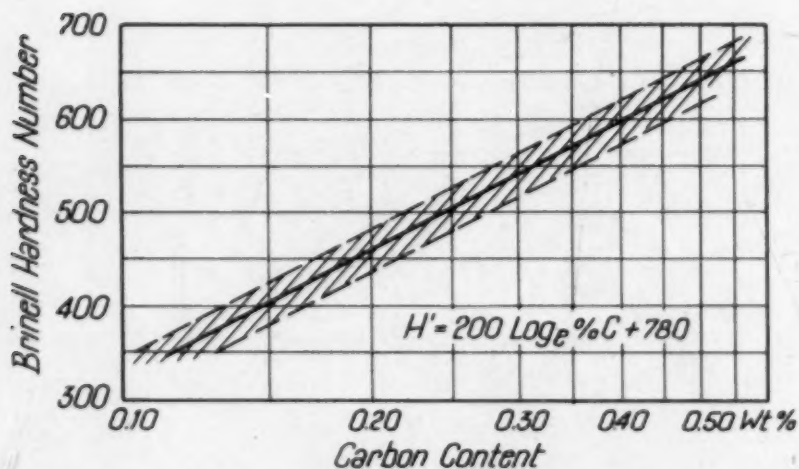


Fig. 3—A relation of Water-Quenched Hardness to Carbon Content of Steels.

Hardness as a Function of Tensile Strength—Ample information is available establishing the validity of this function over a wide range of hardness and tensile strength for plain carbon and alloy steels. For the purpose of this paper, only that range of hardness and tensile strength generally found in normalized steels has been considered. The data used in Fig. 4 have been taken from the METALS HANDBOOK (7) and appear within these limits to be best approximated by this equation:

$$\text{T.S.} = 465H + 5000, \quad (3)$$

where T.S. = tensile strength in pounds per square inch, and
H = hardness (BHN).

Hardenability as a Function of Tensile Strength—By combining Equations (1), (2), and (3), a general empirical expression relating hardenability and tensile strength for any rate of cooling from the austenitizing temperature has been obtained as shown below:

$$\text{T.S.} = \frac{93,000 \log_e \text{Per Cent Carbon} + 362,700}{A \log_e D_1 + B} + 5000. \quad (4)$$

The constants, A and B, evidently define the rate of cooling after an austenitizing treatment. Therefore, once the constants are de-

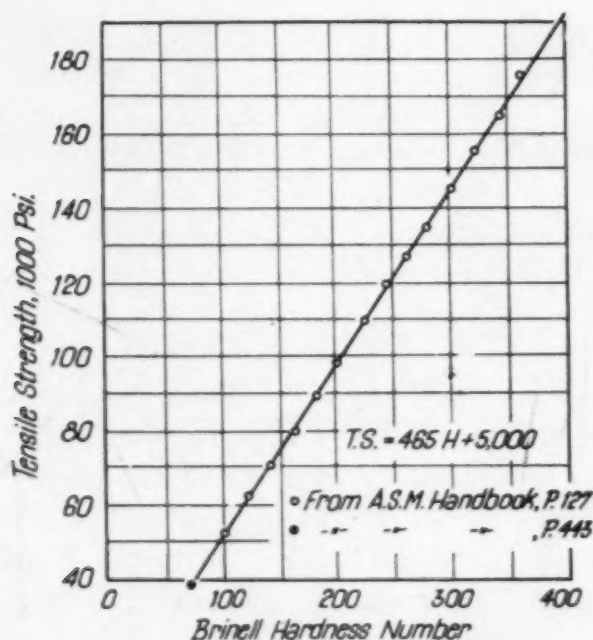


Fig. 4—A Relation of Tensile Strength to Hardness of Steels.

termined for a particular rate of cooling, the tensile strength can then be computed from the hardenability of a steel subjected to this type of heat treatment.

In order to determine the constants, A and B, for steels given a commercial normalizing heat treatment, the data presented in Table I were used. These experimental steels were selected to give a wide range of carbon contents and hardenability values; they represent a variety of steels ranging from the plain carbon to high alloy types. Only the carbon content is indicated; a complete analysis was not performed for reasons of expedience and because the hardenability values were determined experimentally.

Table I
The Hardenability and Normalized Tensile Strength of Some Experimental Steels

Steel No.	Per Cent Carbon	D ₁ (obs.)	T.S. (obs.)	Steel No.	Per Cent Carbon	D ₁ (obs.)	T.S. (obs.)
E-1	0.25	0.55	70,850	E-14	0.35	2.53	105,000
E-2	0.30	0.75	76,700	E-15	0.40	2.65	120,000
E-3	0.36	1.03	89,500	E-16	0.33	3.10	119,000*
E-4	0.35	1.55	90,100	E-17	0.40	3.20	126,500
E-5	0.20	1.76	83,400	E-18	0.27	3.90	119,000
E-6	0.40	1.80	93,500	E-19	0.27	3.94	129,000*
E-7	0.50	1.85	120,000	E-20	0.39	4.18	134,000
E-8	0.28	1.97	95,000	E-21	0.41	4.80	150,000*
E-9	0.43	2.19	116,000*	E-22	0.32	4.85	130,000
E-10	0.31	2.37	107,000*	E-23	0.28	5.50	145,000
E-11	0.29	2.40	95,000	E-24	0.36	5.50	158,000*
E-12	0.32	2.40	102,000*	E-25	0.31	5.80	162,000
E-13	0.38	2.40	108,000				

*These values were converted from normalized hardness data and are considered to be within ± 5 per cent of the actual tensile strength.

The constants were established by applying the method of least square to Equation (4) and the data set forth in Table I. Accordingly, the tensile strength of normalized steels and hardenability have been found to be related as follows:

$$T.S. = \frac{93,000 \log_e \text{Per Cent Carbon} + 362,700}{-0.85 \log_e D_1 + 3.25} + 5000. \quad (5)$$

Curves computed for constant carbon contents according to this equation are shown in Fig. 5. The graph may be conveniently used for obtaining approximate values of normalized tensile strength from hardenability data. The lower limit is set at $D_1 \approx 0.5$ inch in the absence of sufficiently reliable data for very low hardenability values. The upper limit, on the other hand, is restricted to hardenability values for steels which produce little or no martensite upon normalizing.

COMMERCIAL REALIZATION OF THE RELATIONSHIP

Voluminous particulars have been published on the subjects of hardenability and the normalized physical properties of steels. Of all the available information, relatively little has been sufficiently complete to test the validity of Equation (5). Only the data of Jackson and his co-workers (8) and that of Sims and Dahle (9) are entirely satisfactory for this purpose. In the cases where the hardenability values were not determined experimentally, they were computed from the Grossmann hardenability factors (3) for all elements except in the ranges of high silicon and manganese contents where the factors of Crafts and Lamont (10) were employed.

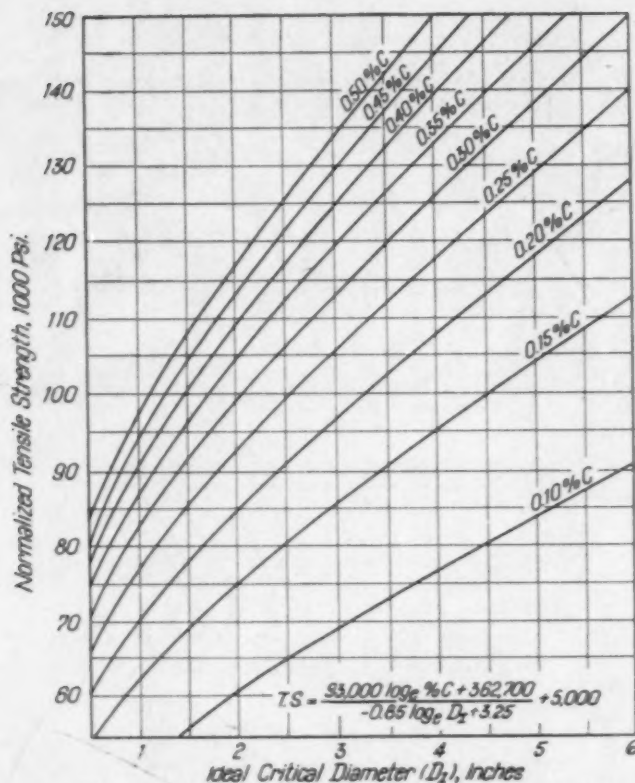


Fig. 5—A Relation of Normalized Tensile Strength to Ideal Critical Diameter (Hardenability) for Constant Carbon Content of Steels.

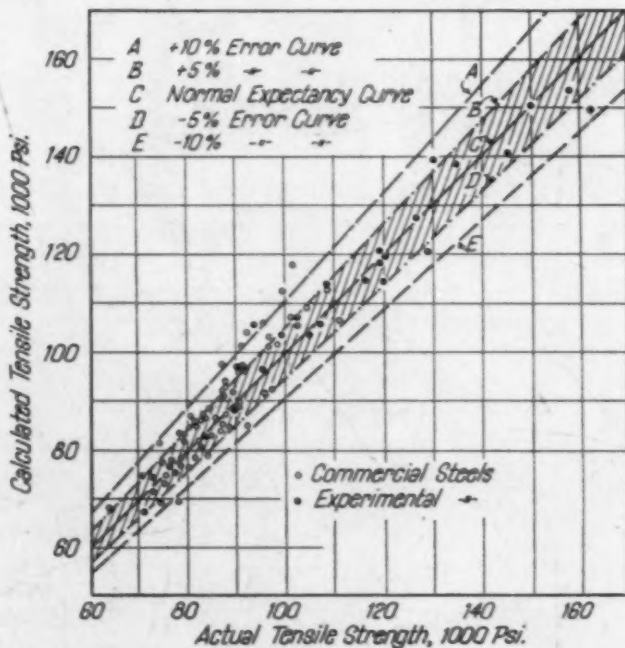


Fig. 6—A Comparison Between Computed and Actual Tensile Strength of Normalized Steels.

Table II
Normalized Silicon-Manganese Steels (8)

Steel No.	C	Mn	Si	S	P	Cu	Ni	Cr	Mo	Al	Grain Size	Dr (obs.)	($\frac{\text{obs.}}{\text{calc.}}$) T.S. (psi.)
224	0.18	0.84	0.66	0.038	0.016	0.23	0.14	0.05	0.02	0.03	2.0	1.80	78,200
227	0.19	1.04	0.67	0.039	0.014	0.23	0.12	0.05	0.02	0.03	1.2	1.85	81,300
230	0.22	1.44	0.69	0.037	0.013	0.20	0.09	0.06	0.02	0.03	1.6	2.70	91,000
233	0.25	0.86	0.69	0.039	0.014	0.22	0.10	0.05	0.02	0.03	1.7	1.95	84,500
239	0.29	1.40	0.70	0.040	0.013	0.23	0.10	0.05	0.02	0.03	2.2	2.20	96,000
236	0.28	0.92	0.71	0.040	0.013	0.22	0.10	0.05	0.02	0.03	1.6	2.00	87,300
247	0.35	1.06	0.72	0.040	0.013	0.22	0.10	0.05	0.02	0.03	1.6	2.50	99,000
242	0.35	0.85	0.73	0.037	0.013	0.22	0.10	0.06	0.02	0.03	1.9	2.10	95,300
228	0.22	1.04	0.87	0.038	0.014	0.23	0.12	0.05	0.02	0.03	1.6	1.95	83,300
231	0.22	1.48	0.90	0.039	0.013	0.20	0.09	0.06	0.02	0.03	1.9	2.55	96,600
234	0.24	0.86	0.90	0.038	0.013	0.22	0.10	0.05	0.02	0.03	1.7	2.00	87,400
240	0.29	1.40	0.92	0.041	0.013	0.22	0.10	0.05	0.02	0.03	2.3	2.50	101,500
225	0.19	0.81	0.93	0.039	0.014	0.23	0.14	0.05	0.02	0.03	1.4	1.75	78,900
243	0.33	0.86	0.94	0.038	0.013	0.22	0.10	0.05	0.02	0.03	1.8	2.05	96,400
245	0.35	1.07	0.94	0.040	0.012	0.23	0.10	0.05	0.02	0.03	2.4	2.35	100,800
249	0.32	1.49	0.94	0.039	0.012	0.22	0.10	0.06	0.02	0.03	2.2	3.05	107,600
226	0.18	0.86	1.12	0.038	0.015	0.23	0.14	0.05	0.02	0.04	2.3	1.75	83,800
232	0.20	1.50	1.12	0.039	0.014	0.20	0.10	0.06	0.02	0.03	1.9	2.50	95,400
235	0.24	0.88	1.12	0.038	0.012	0.22	0.10	0.05	0.02	0.03	1.7	1.80	90,300
229	0.20	1.07	1.14	0.037	0.012	0.23	0.12	0.05	0.02	0.03	2.4	1.90	86,700
241	0.28	1.40	1.15	0.038	0.013	0.23	0.10	0.05	0.02	0.03	1.8	2.75	100,800
246	0.35	1.07	1.17	0.040	0.013	0.22	0.10	0.05	0.02	0.03	1.5	2.30	103,900
238	0.20	0.93	1.18	0.040	0.012	0.23	0.10	0.05	0.02	0.03	1.6	2.00	90,700
250	0.31	1.50	1.19	0.039	0.012	0.22	0.10	0.05	0.02	0.03	2.8	2.95	110,400

Table III
A Variety of Commercial Normalized Steels

(a) Acid Open Hearth Steels (9)										Grain Size	Dr (Calc.)	T.S. (psi.)	
Steel No.	C	Mn	Si	P	S	Cu	Cr	Ni	Mo			Obs.	Calc.
493	0.21	0.69	0.42	0.030	0.035	0.11	0.05	0.01	0.006	7-8	0.80	74,500	69,100
509	0.24	0.66	0.47	0.046	0.035	0.12	0.04	0.01	0.006	7-8	0.88	75,000	73,200
F-1	0.33	0.67	0.36	0.036	0.036	0.05	0.02	0.04	0.060	6-7	1.20	84,000	88,700
F-2	0.30	0.85	0.37	0.035	0.033	0.04	0.02	0.04	0.051	6-7	1.11	86,500	84,400
(b) Basic Open Hearth Steels (9)										6-7	1.11	74,000	81,400
9551	0.27	0.58	0.46	0.013	0.025	0.15	0.14	0.06	0.016	7	1.24	78,000	83,600
9567	0.27	0.68	0.45	0.019	0.034	0.10	0.12	0.05	0.012	7	1.09	82,500	85,800
0-1	0.32	0.72	0.34	0.017	0.027	0.12	0.05	0.15	0.040	6-7	1.10	80,000	84,100
0-2	0.30	0.77	0.34	0.016	0.029	0.12	0.06	0.17	0.030	8	1.04	83,000	87,900
(c) Acid Electric Steels (9)										8	0.91	80,000	76,300
715	0.27	0.80	0.45	0.027	0.034	0.06	0.05	0.03	0.013	7	1.14	82,500	80,600
724	0.26	0.72	0.39	0.026	0.036	0.08	0.03	0.06	0.009	7	1.05	78,500	76,900
1009	0.26	0.68	0.40	0.034	0.038	0.11	0.06	0.18	0.038	7-8	0.81	75,000	74,200
1025	0.24	0.66	0.39	0.035	0.040	0.11	0.06	0.16	0.034	7-8	2.04	92,000	104,400
1163	0.26	0.68	0.40	0.036	0.037	0.12	0.19	0.43	0.06	8	1.01	84,500	82,400
(d) Basic Electric Steel (9)										8	1.42	87,500	93,900
E-1	0.34	0.88	0.28	0.033	0.035	0.36	0.04	0.08	0.056	8	0.95	81,000	85,900
(e) Converter Acid Electric Steels (Triplex) (9)										8+	1.02	90,000	88,400
H-30	0.30	0.73	0.40	0.029	0.035	0.39	0.04	0.06	0.062	8	1.04	80,500	86,300
H-34	0.34	0.78	0.40	0.033	0.036	0.39	0.04	0.06	0.006	8	0.84	74,500	77,000
H-38	0.33	0.58	0.31	0.023	0.041	0.43	0.03	0.08	0.007	7-8	0.91	76,500	77,400
H-42	0.35	0.73	0.35	0.059	0.047	0.38	0.03	0.10	0.005	7	0.71	72,500	72,100
(f) Converter Steels (9)										7	0.71	77,000	75,000
1	0.26	0.77	0.55	0.034	0.041	0.19	0.04	0.09	0.008	8	1.26	87,500	92,400
2	0.26	0.66	0.38	0.032	0.024	0.09	0.04	0.06	0.018	8	1.07	85,500	86,400
3	0.27	0.65	0.37	0.038	0.026	0.09	0.05	0.06	0.014	7-8	0.86	81,000	78,300
4	0.26	0.50	0.32	0.033	0.028	0.14	0.03	0.05	0.007	7-8	0.44	63,600	68,500
A	0.29	0.50	0.33	0.040	0.031	0.09	0.02	0.085	0.008	7-8	0.60	72,800	74,600
B	0.33	0.85	0.43	0.043	0.035	0.09	0.02	0.072	0.006	7-8	0.75	78,000	77,500
C	0.33	0.64	0.39	0.049	0.038	0.10	0.03	0.079	0.007	7-8	1.02	90,000	88,400
D	0.29	0.73	0.36	0.044	0.035	0.09	0.03	0.077	0.014	7-8	0.86	81,000	78,300
(g) Structural Steels (11)*										7-8	0.44	63,600	68,500
17	0.30	0.42	0.07	0.02	0.04	0.09	0.04	0.06	0.006	7-8	0.60	72,800	74,600
18	0.32	0.44	0.47	0.02	0.04	0.09	0.04	0.06	0.006	7-8	0.75	78,000	77,500
19	0.31	0.46	0.96	0.02	0.04	0.09	0.05	0.06	0.007	7-8	1.02	88,200	84,500
20	0.32	0.49	1.53	0.02	0.04	0.09	0.03	0.05	0.007	7-8	0.66	70,400	74,700
21	0.30	0.76	0.06	0.02	0.04	0.09	0.02	0.085	0.008	7-8	0.98	78,800	82,600
22	0.31	0.89	0.54	0.02	0.04	0.09	0.02	0.072	0.008	7-8	1.35	88,800	91,500
23	0.33	0.94	1.10	0.02	0.04	0.09	0.03	0.079	0.018	7-8	1.71	98,000	101,000
24	0.36	0.94	1.65	0.02	0.05	0.09	0.03	0.077	0.014	7-8	1.08	81,200	84,800
25	0.31	1.34	0.08	0.03	0.05	0.09	0.03	0.077	0.014	7-8	1.37	86,800	91,200
26	0.32	1.38	0.48	0.02	0.04	0.09	0.03	0.077	0.014	7-8	1.63	91,200	95,500
27	0.32	1.31	0.93	0.02	0.04	0.09	0.03	0.077	0.014	7-8	1.17	87,600	86,700
28	0.31	1.50	0.08	0.02	0.04	0.09	0.03	0.077	0.014	7-8	1.58	90,000	95,500
29	0.33	1.47	0.61	0.02	0.03	0.09	0.03	0.077	0.014	7-8	1.58	90,000	95,500

*Grain size assumed.

The calculated normalized tensile strength values agree fairly well with those observed, as brought out in Tables II and III. A comparison between the calculated and observed tensile strength is illustrated in Fig. 6. It is clear from this figure that the variation is rarely more than ± 10 per cent; in the majority of cases, it is less than ± 5 per cent of the normal expectancy value.

It is important to note that the carbon content influences the tensile strength greatly as seen in Fig. 5; hence, a slight error in the carbon content will introduce a considerable disparity between the computed and observed tensile values. Moreover, a computed value which is unreasonably higher than observed may indicate that the maximum effect of the alloying elements has not been fully developed. On the other hand, a very low value may be often attributed to incomplete analysis.

SUMMARY

Three independent and empirical relationships have been established mathematically as follows:

1. Hardenability as a function of the hardness ratio for any definite rate of cooling from the austenitizing temperature:

$$\frac{H'}{H} = A \log_e D_1 + B. \quad (1)$$

2. Water-quenched hardness as a function of the carbon content:

$$H' = 200 \log_e \text{Per Cent Carbon} + 780. \quad (2)$$

3. Hardness as a function of tensile strength:

$$\text{T.S.} = 465H + 5000. \quad (3)$$

By combining these three functions, the tensile strength of steels becomes related to the hardenability according to this general equation:

$$\text{T.S.} = \frac{93,000 \log_e \text{Per Cent Carbon} + 362,700}{A \log_e D_1 + B} + 5000 \quad (4)$$

The constants, A and B, depend on the rate of cooling from the austenitizing temperature. In the case of a normalizing heat treatment, the constants have the following values:

$$A = -0.85, \text{ and} \\ B = 3.25.$$

The validity of this equation has been confirmed in a wide variety of commercially normalized steels. This relationship will assist in

developing new steel compositions with desired combinations of hardenability and normalized tensile properties by using the alloying elements efficiently and economically.

ACKNOWLEDGMENT

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ANNEALING STUDIES ON COLD-ROLLED IRON AND IRON BINARY ALLOYS

BY CHARLES R. AUSTIN, L. A. LUINI, AND R. W. LINDSAY

Abstract

Investigation of the behavior of a group of iron binary alloys has been extended to include the study of the response of these alloys to strain hardening by cold rolling and the behavior of the cold-rolled alloys during annealing. The data presented are for the alloys of iron with nickel, chromium, cobalt, silicon, manganese and molybdenum. These alloys were reduced in thickness by 5, 20, 40, 75 and 90 per cent and the cold-rolled alloys were annealed subsequently at temperatures ranging from 625 to 1400 degrees Fahr. (330 to 760 degrees Cent.) employing a constant annealing period of 1 hour at each temperature. The effect of time as a variable was studied at 900 degrees Fahr. (480 degrees Cent.). Comparison studies were made on unalloyed iron in all instances. Changes during cold working and annealing were followed by means of Vickers hardness measurements and micro-examination.

The results on unalloyed iron were in agreement with other investigations upon the subject of cold working and annealing of metals and alloys, as regards the effect of amount of deformation and the processes of recovery, grain growth and recrystallization.

In most cases the binary alloys behaved similarly to unalloyed iron in their response to cold rolling. There were significant differences in behavior during annealing, with molybdenum, chromium and manganese being effective in displacing the softening of the cold-worked samples to higher temperatures in comparison to the softening temperature of iron.

THE mechanism of strain hardening in metals and their alloys, and the removal of strain hardening by annealing, has always stimulated interest among metallurgists. It is recognized that the

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number of factors involved in a study of this subject is several and makes such studies rather complex and extensive. These factors would include the prior history and original structure of the material involved, the type and amount of deformation to which it was subjected, the temperature and time of annealing and the chemical composition of the material.

Numerous investigators have concerned themselves with these factors in most of the common metals and alloys and pure iron has received its share of attention. However, it is striking that iron has been neglected in one important respect, that is the effect of second element additions on the strain hardening characteristics and response to annealing.

The need for such data is rather obvious. In ferrous materials the matrix quite frequently consists of ferrite which often contains other elements in solid solution. Knowledge of the behavior of iron binary solid solution alloys would be of material benefit in several ways. For example, many ferrous materials are shaped and formed by cold working operations and it is possible that the strain hardening capacity of ferrite and its capacity for further deformation may be affected by the presence of other elements. Furthermore, in commercial fabricating operations it is often necessary to subject the cold-worked material to annealing and it is of importance to know the manner in which elements in solid solution in ferrite affect the various aspects of this annealing process. At other times this ferritic phase is subjected to a combination of stress and elevated temperature and again the effects of added elements in solid solution in ferrite have received meager quantitative study.

Accordingly the present work was undertaken to investigate the strain hardening characteristics of unalloyed iron and its subsequent softening during annealing and to ascertain the effect upon these processes of a second element in solid solution in the iron.

REVIEW OF LITERATURE

The literature on cold working and annealing of metals and alloys is quite voluminous. This review will be confined to iron and iron alloys, referring to results on other metals and their alloys at points where they apply to the presentation.

A large share of attention has been devoted to the cold working and annealing of iron of a more or less high degree of purity. An

excellent bibliography on this subject has been given by Kaiser and Taylor (1).¹ Kenyon (2) studied the behavior of Armco iron on cold rolling and found that the hardness increased at a decreasing rate up to 13 per cent reduction in thickness at which point the hardness had increased from Rockwell B-32 to B-65. Butler (3) has shown that the first slight amount of cold work raises the tensile strength and hardness of iron and iron alloys at a rapid rate. This is followed by a lesser and rather uniform increase and with still further degree of cold deformation these properties increase at a more rapid rate.

A concise summary of studies of the effect of cold work on the properties of iron is given in the METALS HANDBOOK (4). This summary includes also some references to investigations on the annealing of cold-worked iron.

Oberhoffer and Oertel (5), (6) carried out an extensive investigation on the recrystallization of cold-worked iron. They found that the temperature at which recrystallization began depended chiefly on the amount of previous deformation. The greater the amount of deformation, the lower was the temperature for the beginning of recrystallization. The change in Brinell hardness could not be shown to be related to changes in microstructure and appeared to be independent of grain size. The specimen compressed 5 per cent showed substantial softening after annealing at elevated temperatures, without any visible structural change. These investigators constructed a three-dimensional diagram showing the relation between amount of deformation, annealing temperature, and resulting grain size.

Tammann and Moritz (7) found that the increase in indentation hardness of electrolytic iron caused by cold working was removed partially by annealing at temperatures between 500 and 600 degrees Cent. (930 and 1110 degrees Fahr.), but annealing at 800 degrees Cent. (1470 degrees Fahr.) or above was necessary for complete softening.

Sauerwald and Globig (8) showed that it was possible to soften cold-worked iron without the occurrence of recrystallization. They found that with low annealing temperatures, recovery is the principal process involved. The rate of softening decreased with time and softening was not complete even after long times. Recrystallization occurred at higher temperatures and gave rise to a sudden increase in the rate of softening.

¹The figures appearing in parentheses pertain to the references appended to this paper.

Pomp and Niebch (9) worked with Krupp soft iron and found that samples stretched 5 per cent in tension softened only by recovery.

Quite frequently with low amounts of deformation exceptionally coarse-sized grains develop upon annealing. This was shown in earlier investigations by Sauveur (10) and by Chappell (11). In investigating the recrystallization of commercial iron, Oberhoffer and Jungbluth (12) found this condition to exist after 10 per cent deformation followed by annealing at 700 to 800 degrees Cent. (1290 to 1470 degrees Fahr.). This study was repeated by Oberhoffer and Oertel (5) using electrolytic iron and in this material these con-

Table I
Softening Temperatures of Iron Binary Alloys After 90 Per Cent Reduction by Rolling
(After Tammann (14))

Material	Softening Temperature in Degrees Cent.
Fe (Electrolytic)	520
4 Atomic per cent Al	600
2 Atomic per cent Si	600
2 Atomic per cent V	620
4 Atomic per cent Cr	700
4 Atomic per cent Co	640
4 Atomic per cent Ni	600
2 Atomic per cent Mo	750
1 Atomic per cent W	760

ditions did not lead to a coarse grain size. Kaiser and Taylor (1) showed that the temperature at which abnormal grain growth occurred is independent of the type of deformation. They indicate also that variations between the behavior of commercial iron and electrolytic iron may be because different materials require different critical amounts of deformation. Pomp and Niebch (9) found large grains to be developed in some of their samples and attributed this to recrystallization, although it would seem that such coarsening after limited cold deformation may relate to true grain growth without recrystallization.

Very little work has been done on the effects of added elements on the annealing characteristics of iron. Tammann (13), (14) has published probably the most extensive piece of work on this subject, investigating the effects of additions of aluminum, silicon, vanadium, chromium, cobalt, nickel, molybdenum and tungsten to electrolytic iron. The samples were reduced 90 per cent by rolling prior to annealing. His data on iron binary alloys is reproduced in Table I.

Butler (3) has shown that additions of 2 per cent of nickel, chromium, and molybdenum raised the temperature at which recrystallization began by 20, 50, and 200 degrees Cent. respectively.

EXPERIMENTAL PROCEDURE

The method of preparation of these alloys has been discussed in a previous publication (15) and the analyses are listed in Table II. In all instances, the percentage of added element was less than the limit of solid solubility in alpha iron and hence the investigation deals with alloyed ferrites.

Table II
Chemical Composition of Alloys Investigated

Alloy	Weight Per Cent Alloying Element								S
	C	Cr	Co	Ni	Mn	Mo	Si	P	
Pure Iron	0.02	0.003	0.005	0.032	0.03	0.004	0.003	0.010	0.013
Chromium	0.03	0.45	0.005	0.032	0.05	0.004	0.012	0.011	0.011
	0.02	0.99	0.005	0.034	0.03	0.004	0.004	0.010	0.013
	0.03	4.83	0.005	0.023	0.03	0.004	0.008	0.012	0.015
Cobalt	0.02	0.003	0.52	0.037	0.03	0.004	0.004	0.012	0.014
	0.02	0.003	1.00	0.043	0.05	0.004	0.004	0.010	0.019
	0.02	0.003	5.08	0.08	0.03	0.004	0.004	0.012	0.015
Nickel	0.03	0.003	0.005	0.57	0.03	0.004	0.004	0.010	0.011
	0.02	0.003	0.005	1.15	0.03	0.004	0.004	0.010	0.015
	0.02	0.003	0.005	4.83	0.03	0.004	0.004	0.010	0.016
Manganese	0.02	0.003	0.005	0.032	0.69	0.004	0.004	0.010	0.014
	0.06	0.003	0.005	0.030	1.33	0.004	0.004	0.011	0.020
Molybdenum	0.03	0.003	0.005	0.054	0.03	0.11	0.004	0.010	0.014
	0.03	0.003	0.005	0.023	0.03	0.54	0.004	0.010	0.014
	0.04	0.003	0.005	0.016	0.03	1.50	0.004	0.011	0.014
Silicon	0.03	0.003	0.006	0.033	0.03	0.004	0.22	0.012	0.015
	0.02	0.004	0.005	0.032	0.03	0.004	0.59	0.011	0.016
	0.02	0.003	0.005	0.055	0.03	0.004	1.21	0.010	0.012

Cold Rolling—The alloys as-received, in the form of bar stock of $\frac{5}{16}$ -inch diameter, were cold-rolled to a thickness of 0.223 inch. The materials in this condition were subjected to a softening anneal consisting of heating to 975 degrees Cent. (1785 degrees Fahr.) for 1 hour, followed by furnace cooling to 650 degrees Cent. (1200 degrees Fahr.) and holding for 20 hours before furnace cooling to room temperature. All heat treatments in the investigation were conducted in a vacuum of 100 microns or better with temperatures controlled to ± 1 degree Cent. The selection of the pretreatment resulting in a dead soft condition of the alloy was dependent on the experimental observations already cited.

The softened alloys were given reductions in thickness of 5, 20, 40, 75, and 90 per cent by flat cold rolling. The thickness was reduced approximately 0.015 inch per pass. A small length of the dead soft material taken from each alloy was considered as representative of the unworked condition. Additional small lengths were removed after each reduction. The progress of hardening by cold rolling was followed by means of the Vickers hardness test employing a 10-kilogram load applied for 30 seconds. Hardness readings on the rolled pieces were taken on the rolled faces.

Annealing—Specimens of $\frac{1}{4}$ -inch length were cut from the alloys in the various cold-worked conditions and annealed for 1 hour at different temperatures followed by furnace cooling to room temperature. The annealing temperatures used covered a range from 330 to 870 degrees Cent. (625 to 1600 degrees Fahr.). Annealing curves were plotted to relate changes in Vickers hardness to annealing temperature and the changes in hardness were correlated with changes in microstructure.

The effect of time at temperature was studied by annealing specimens at 480 degrees Cent. (900 degrees Fahr.) for various periods up to 20 hours. This phase of the investigation was carried out on a separate set of specimens and served the purpose of studying an important variable. The reason for selection of this particular temperature will be apparent in a later section.

Grain size determinations were made on certain of the alloys using a micrometer eyepiece which gave readings in A.S.T.M. grain size numbers. An especially comprehensive microscopic study was carried out on the unalloyed iron specimens after each annealing treatment and the grain size was determined on each sample.

STUDIES ON THE UNALLOYED IRON

It is felt that the presentation and discussion of the results might be clarified if the behavior of the unalloyed iron was separated from that of the alloys. A comparison between the behavior of the iron and that of the alloys will be made in the conclusions.

The progress of work hardening by cold rolling of the unalloyed iron is shown in Fig. 1 where the increase of hardness is plotted as a function of the percentage reduction in thickness during rolling. It is to be noted that the iron shows an appreciable increase in hardness during the initial stages of reduction, but this increase becomes

less with heavier reductions so that around 40 per cent reduction the curve tends to become parallel to the horizontal axis. Finally the curve turns sharply upward again from 75 to 90 per cent reduction in thickness. This is the conventional method of plotting which frequently exhibits an inflection at the higher reductions per cent, as usually calculated. It is interesting to note that the form of the curve is similar to the stress-strain curves of various metal and alloys tested in tension, wherein the average true stress is plotted as a function of the reduction of area based on the original area (22). This latter variable was similarly computed to provide the data for the abscissa used in Fig. 1.

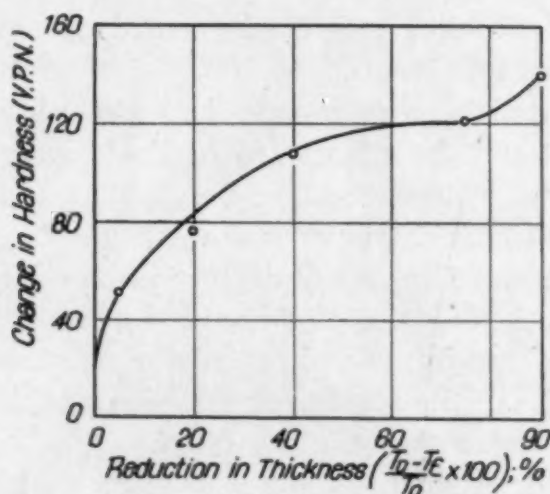


Fig. 1—The Relation Between the Degree of Cold Deformation as Measured by Reduction in Thickness on Rolling and the Vickers Pyramid Hardness for the Commercially Pure Iron.

The microstructures corresponding to various stages of rolling are shown in Fig. 2. These indicate that there is no microstructural evidence of plastic deformation until the reductions in thickness approximate 40 per cent or more. The samples were taken normal to the rolled faces so that the entire thickness from one rolled face to the other was examined.

Samples representing each degree of cold rolling were annealed for 1 hour at 625, 800, 900, 1000, 1200, 1300, 1400, and 1600 degrees Fahr., a new set of samples being annealed at each temperature. The progress of softening with increase of annealing temperature is shown for each of the five reductions of thickness in Fig. 3. Extensive microscopic studies were made on these annealed specimens

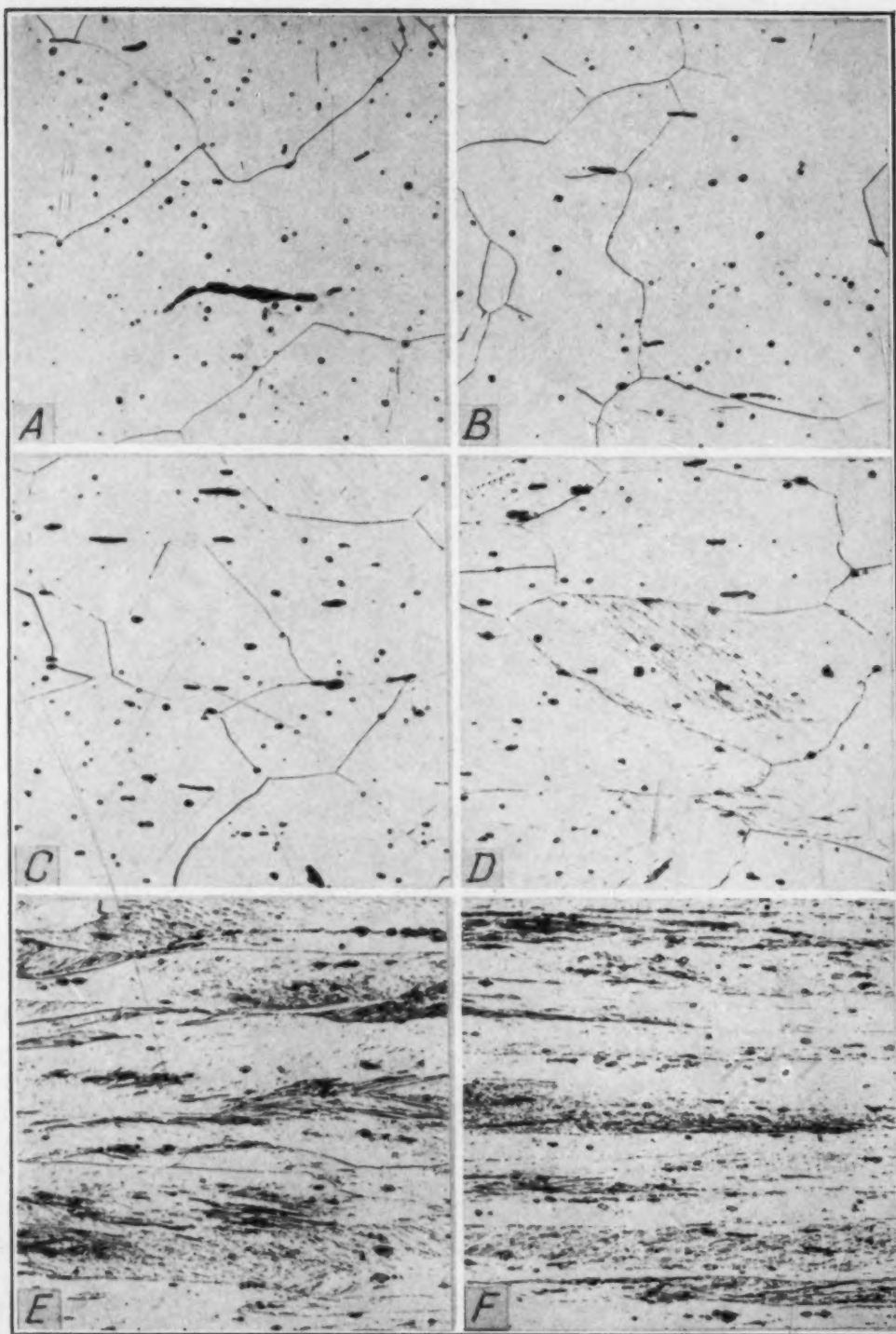


Fig. 2—Effect of Cold Reduction by Rolling on the Structure of the Fully Annealed Commercially Pure Iron. Etched in 4 Per Cent Nital. $\times 100$.

A. Annealed.

B. 5 Per Cent Reduction.

C. 20 Per Cent Reduction.

D. 40 Per Cent Reduction.

E. 75 Per Cent Reduction.

F. 90 Per Cent Reduction.

and certain representative ones were photographed to illustrate changes in structure with relation to changes of hardness. Photomicrographs of these structures are included as Figs. 4 to 8. In conjunction with the microscopic examination, grain size determinations were made before and after the annealing treatments. Thus it was possible to ascertain the grain size during recrystallization, the

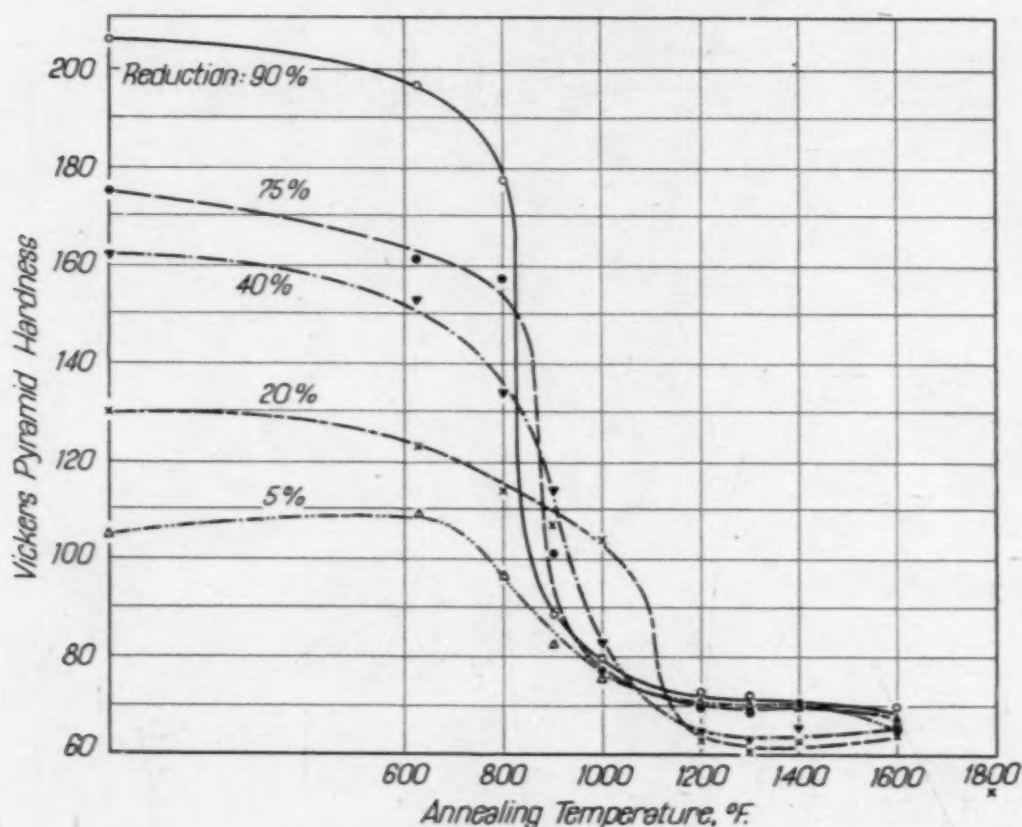


Fig. 3—Relation Between Annealing Temperature and Vickers Pyramid Hardness for the Commercially Pure Iron Reduced 5 to 90 Per Cent in Thickness by Cold Rolling.

grain size just after recrystallization was complete, and changes in grain size as a consequence of grain growth. Data on these studies are tabulated in Table III together with the corresponding Vickers hardness and remarks concerning the progress of structural changes.

It will be noted in the column on comments on microstructure that uncertainty occasionally obtains as to whether recrystallization has been effected. This is dependent on the fact that with relatively low reduction per cent, grain growth can occur by boundary migration without the inception of true recrystallization. Furthermore, recrystallization is also accompanied by grain growth and the prog-

ress of this latter phenomenon is indicated by the column showing grain size after heat treatment.

The relation between reduction in thickness, annealing temperature, and A.S.T.M. grain size may be presented in a clear manner on a three-dimensional type of diagram. Such a diagram has been constructed in Fig. 9. The shaded areas under the curves relating

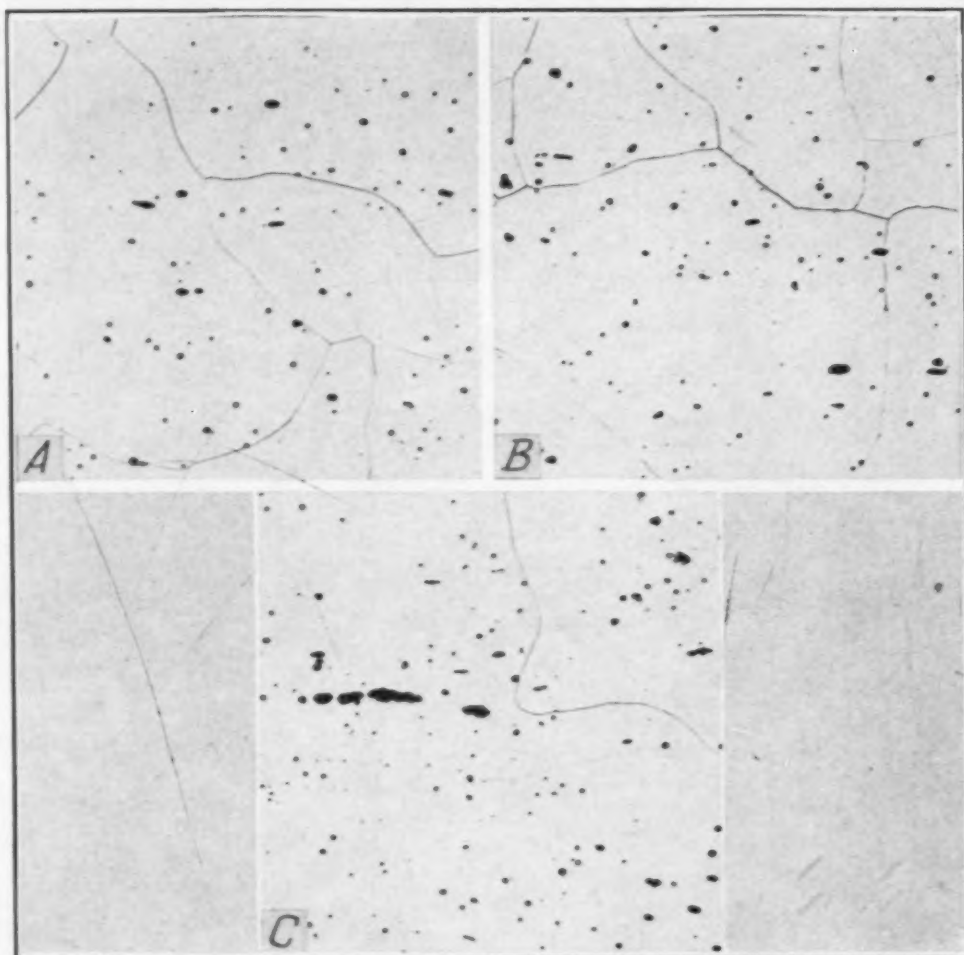


Fig. 4—Effect of Annealing for 1 Hour at Various Temperatures on the Microstructure of Commercially Pure Iron After Reducing 5 Per Cent in Thickness by Cold Rolling. Etched in 4 Per Cent Nital. $\times 100$.

A. 800 Degrees Fahr. B. 1200 Degrees Fahr. C. 1600 Degrees Fahr.

grain size to annealing temperature represent the range of temperature in which recrystallization may be assumed to occur. The height of the curve at the low end of this shaded range is indicative of the initial grain size of new recrystallized grains, while the height of the curve at any higher temperature within this recrystallization range is evidence of the growth of these new grains. Continuations of these curves beyond the shaded areas illustrate grain growth of the

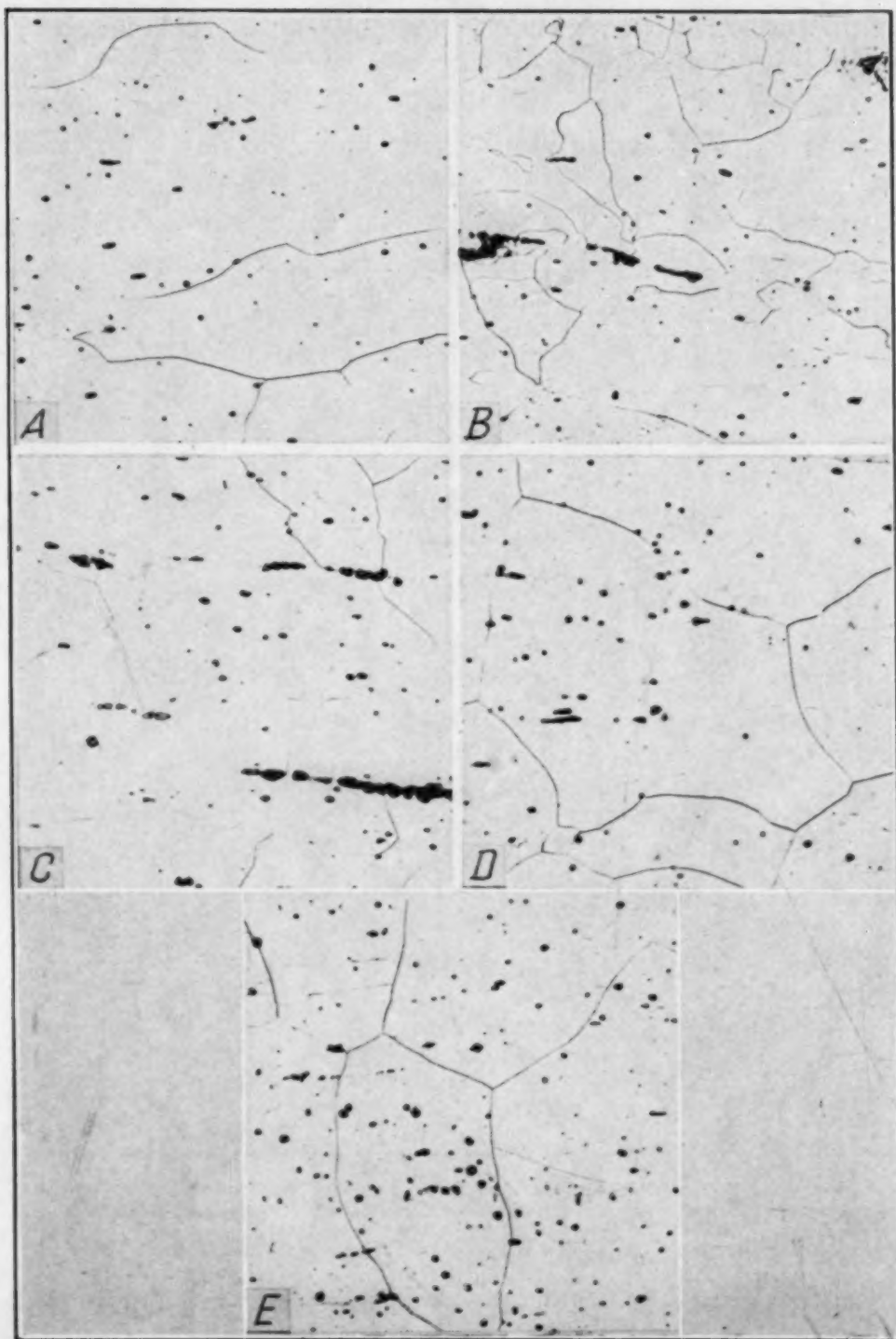


Fig. 5—Effect of Annealing for 1 Hour at Various Temperatures on the Microstructure of Commercially Pure Iron After Reducing 20 Per Cent in Thickness by Cold Rolling. Etched in 4 Per Cent Nital. $\times 100$.
A. 800 Degrees Fahr. B. 1000 Degrees Fahr. C. 1200 Degrees Fahr.
D. 1300 Degrees Fahr. E. 1600 Degrees Fahr.

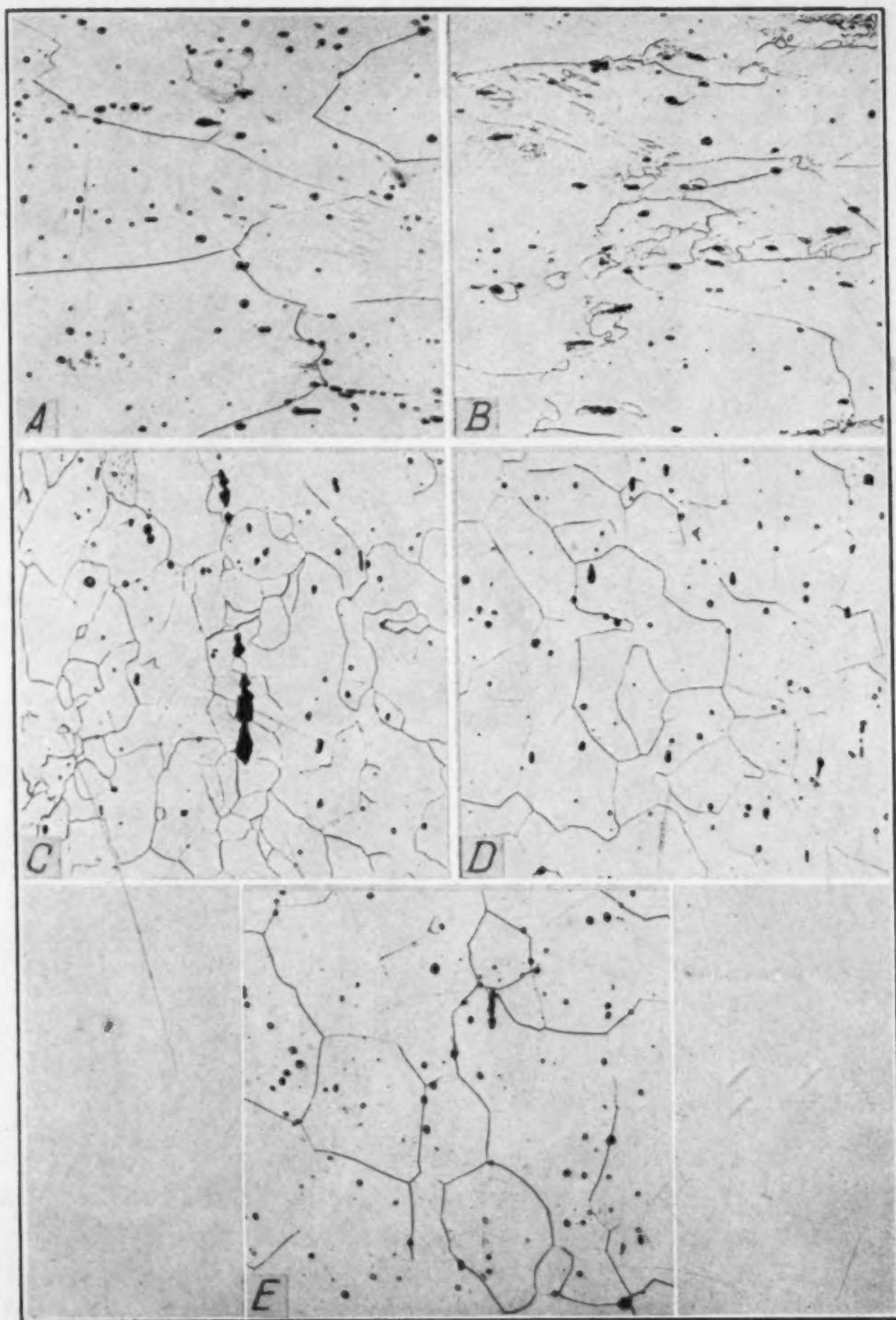


Fig. 6—Effect of Annealing for 1 Hour at Various Temperatures on the Microstructure of Commercially Pure Iron After Reducing 40 Per Cent in Thickness by Cold Rolling. Etched in 4 Per Cent Nital. $\times 100$.
A. 800 Degrees Fahr. B. 900 Degrees Fahr. C. 1000 Degrees Fahr.
D. 1200 Degrees Fahr. E. 1600 Degrees Fahr.

Table III
Details of A.S.T.M. Grain Size, Vickers Pyramid Hardness and Microstructural Characteristics Relating to Commercially Pure Iron Reduced Varying Amounts by Rolling, and Annealed for 1 Hour at the Indicated Temperature

Annealing Temperature	Reduction in Thickness	Grain Size After Heat Treatment	V P N Hardness	Comments on Microstructure
625	5	1-2	109	No change from structure observed after cold rolling.
	20	1-2	123	
	40	1-2	153	
	75	Distorted	161	
	90	Distorted	197	
800	5	1-2	97	No change
	20	1-2	114	No change
	40	1-2	134	No change
	75	Distorted	157	Incipient recrystallization
	90	Distorted	178	Incipient recrystallization
900	5	1-2	83	No change
	20	1-2	107	No change
	40	1-2	114	Incipient recrystallization
	75	7½	101	Largely recrystallized
	90	8	89	Completely recrystallized
1000	5	1-2	77	No change
	20	1-2	104	Incipient recrystallization
	40	5-7	83	Recrystallized
	75	6-7	76	Completely recrystallized
	90	7-8	80	Completely recrystallized
1200	5	1-2	71	No change
	20	2	64	Not clear
	40	5	65	Recrystallization complete
	75	6	71	Recrystallization complete
	90	6	73	Recrystallization complete
1300	5	1-2	72	No change
	20	1-2	61	Not clear
	40	4	63	Recrystallized
	75	5½	69	Recrystallized
	90	6	71	Recrystallized
1400	5	1-2	70	No change
	20	2	63	Not clear
	40	4	66	Recrystallized
	75	5	70	Recrystallized
	90	5½	68	Recrystallized
1600	5	1	66	Grain growth
	20	1-2	67	Not clear
	40	3	66	Recrystallized
	75	4	71	Recrystallized
	90	4	65	Recrystallized

structure, after recrystallization has been completed, at higher annealing temperatures. No recrystallization is indicated for the specimens reduced 5 per cent in thickness and yet at 870 degrees Cent. a structure of exceptionally large-sized grains is developed. The significance of this will be brought out in the discussion.

The three-dimensional diagram shows also the grain sizes developed at any given annealing temperature, above that at which the structure has recrystallized, as a function of degree of cold work. The dotted curve drawn in on the base of the diagram illustrates the interdependence of recrystallization temperature and amount of cold

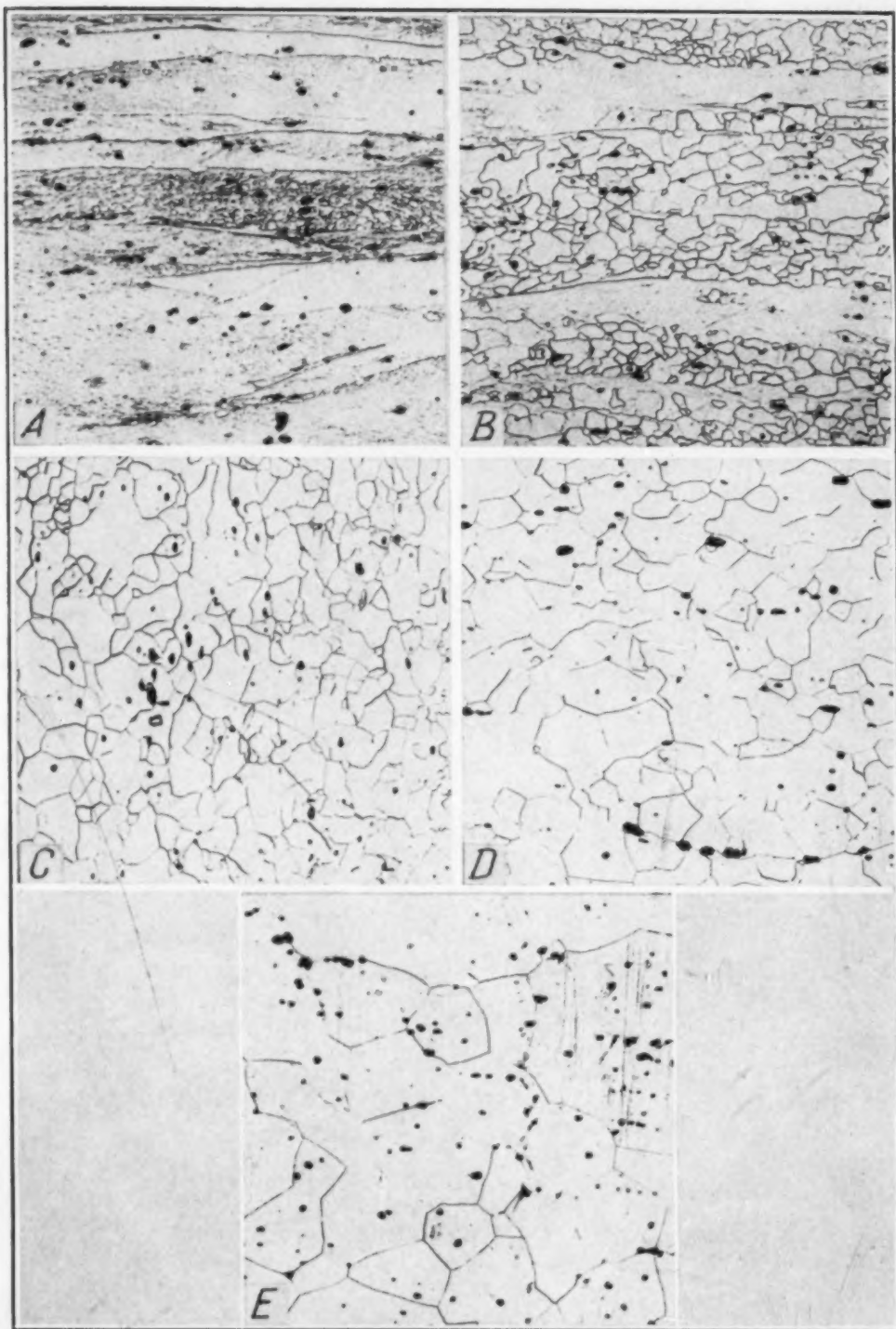


Fig. 7—Effect of Annealing for 1 Hour at Various Temperatures on the Microstructure of Commercially Pure Iron After Reducing 75 Per Cent in Thickness by Cold Rolling. Etched in 4 Per Cent Nital. $\times 100$.

A. 800 Degrees Fahr. B. 900 Degrees Fahr. C. 1000 Degrees Fahr.
D. 1200 Degrees Fahr. E. 1600 Degrees Fahr.

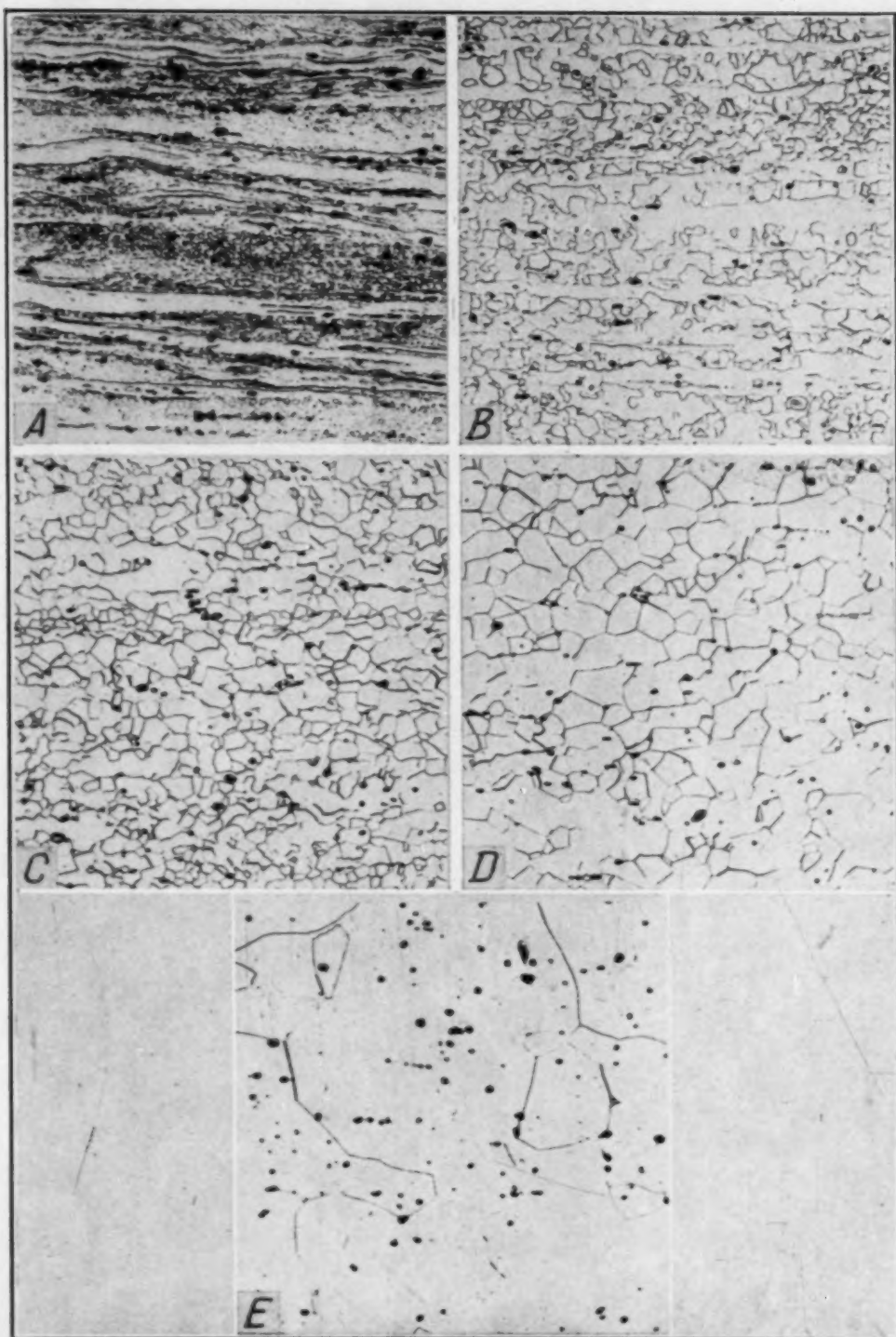


Fig. 8—Effect of Annealing for 1 Hour at Various Temperatures on the Micro-structure of Commercially Pure Iron After Reducing 90 Per Cent in Thickness by Cold Rolling. Etched in 4 Per Cent Nital. $\times 100$.
A. 800 Degrees Fahr. B. 900 Degrees Fahr. C. 1000 Degrees Fahr.
D. 1200 Degrees Fahr. E. 1600 Degrees Fahr.

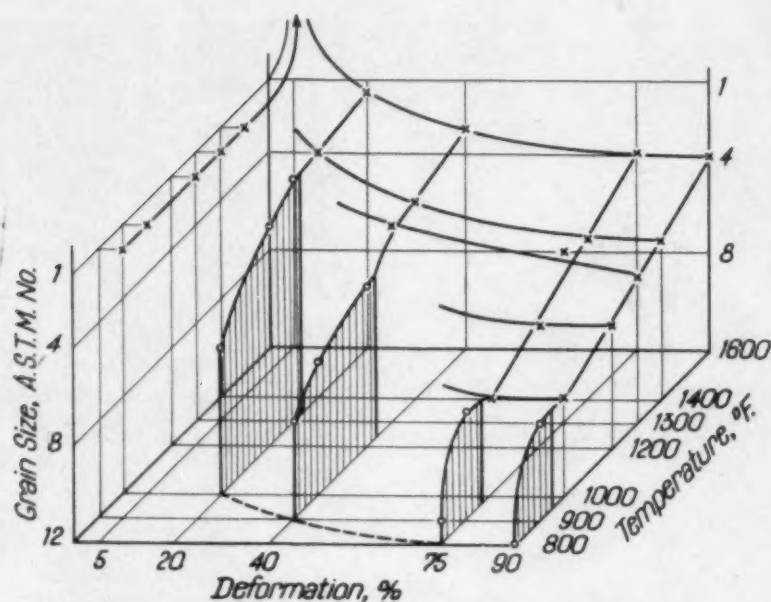


Fig. 9—Relation Between the Effect of 1 Hour Anneal at Temperatures Varying from 800 to 1600 Degrees Fahr. and the A.S.T.M. Grain Size of the Unalloyed Iron, Following Cold Reduction by Rolling 5 to 90 Per Cent.

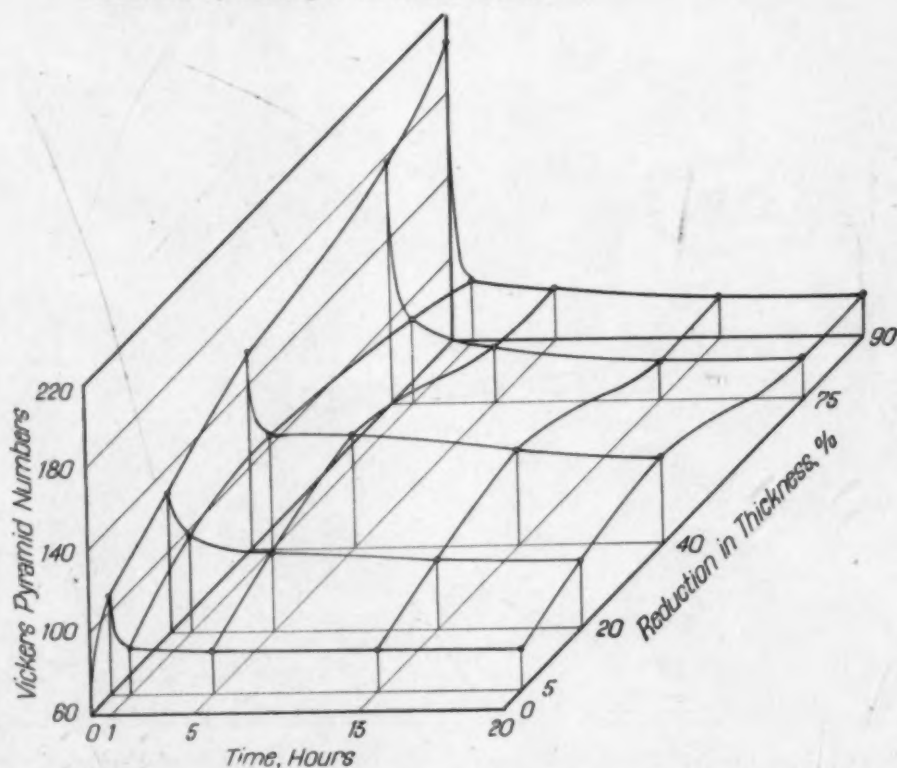


Fig. 10—Relation Between Time of Annealing (1 to 20 Hours) at 900 Degrees Fahr. and the Vickers Pyramid Hardness of the Unalloyed Iron Following Cold Reduction by Rolling 5 to 90 Per Cent.

work. This diagram embraces many corroborations of previously published data on annealing of cold-worked metals (4).

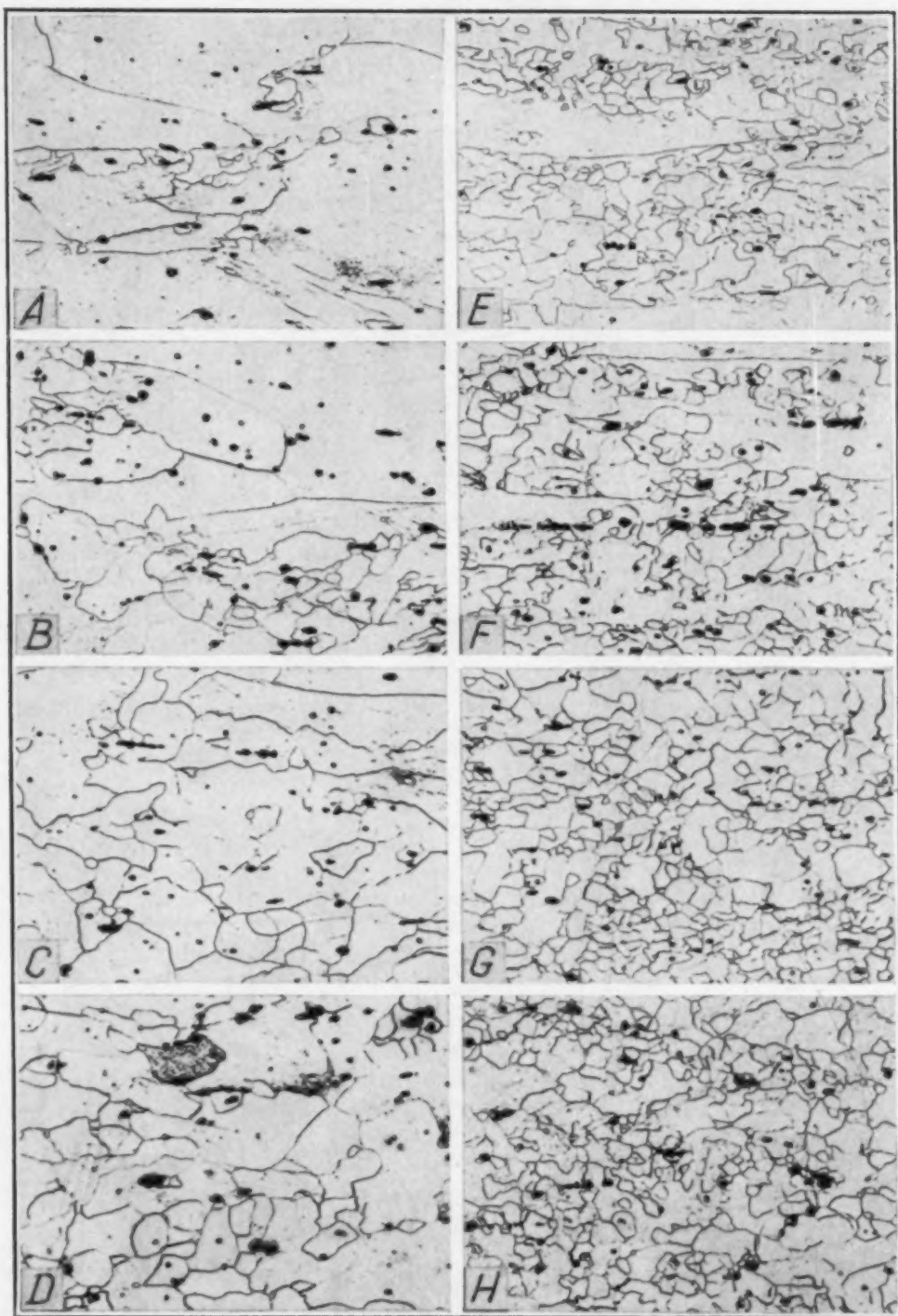


Fig. 11—Effect of Time of Annealing on the Microstructure of Commercially Pure Iron After Cold Rolling 40 and 75 Per Cent Reduction in Thickness. Etched in 4 Per Cent Nital. $\times 100$. Annealing Temperature 900 Degrees Fahr.
40 Per Cent. A. 1 Hour. B. 5 Hours. C. 13 Hours. D. 20 Hours.
75 Per Cent. E. 1 Hour. F. 5 Hours. G. 13 Hours. H. 20 Hours.

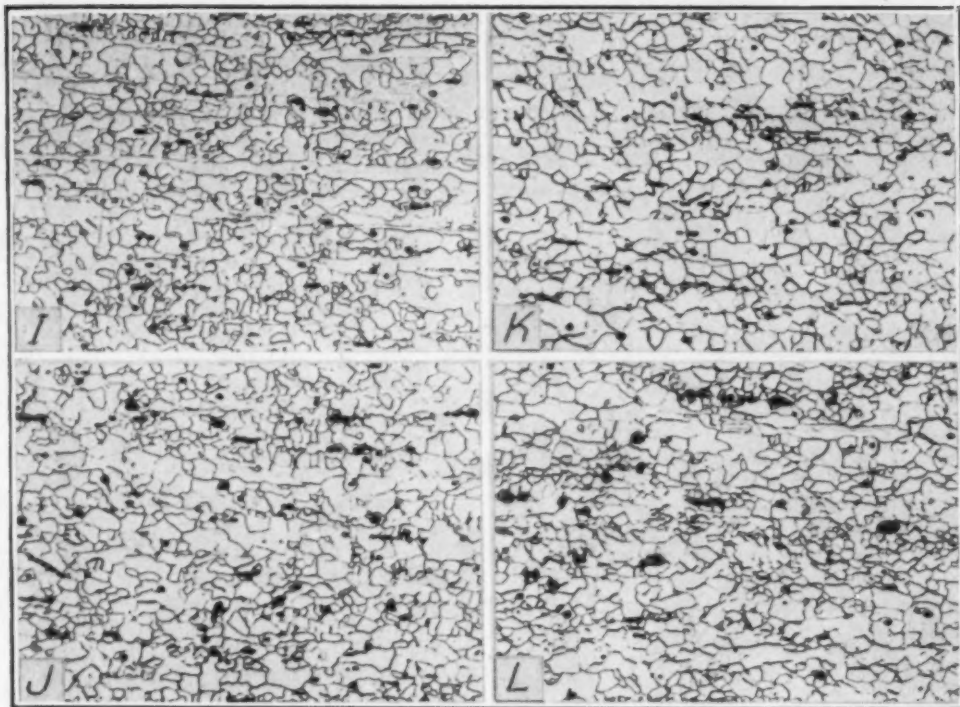


Fig. 11 (cont.)—Effect of Time of Annealing on the Microstructure of Commercially Pure Iron After Cold Rolling 90 Per Cent Reduction in Thickness. Etched in 4 Per Cent Nital. $\times 100$, 90 Per Cent. I. 1 Hour. J. 5 Hours. K. 13 Hours. L. 20 Hours. Annealing Temperature 900 Degrees Fahr.

A study of the effect of time on the softening of cold-worked iron was made at 480 degrees Cent. (900 degrees Fahr.). This temperature was chosen because it was evident from the foregoing data that softening would be sufficiently rapid to be followed in a reasonable period of time and yet not too rapid to be studied conveniently. It will be noted also that this temperature approximates the inflection position on the various hardness-annealing temperature curves shown in Fig. 3. Again the results were plotted on a three-dimensional diagram correlating Vickers hardness, time and per cent reduction of thickness. This diagram is shown in Fig. 10. Inspection of this figure will show that for any annealing time there appears to be a maximum in the curve of hardness versus per cent reduction of thickness, this maximum occurring at 40 per cent reduction. An explanation will be offered for this in the discussion. The photomicrographs in Fig. 11 show the changes in microstructure resulting after annealing for 1, 5, 13, and 20 hours samples reduced 40, 75, and 90 per cent in thickness. The microstructures of the samples as-rolled have been presented already in Fig. 2. Photomicrographs taken of samples reduced 5 per cent and 20 per cent in thickness

were not included since no microstructural changes were noted. This condition could readily be foreseen from the microstructural characteristics already discussed (Figs. 4 and 5).

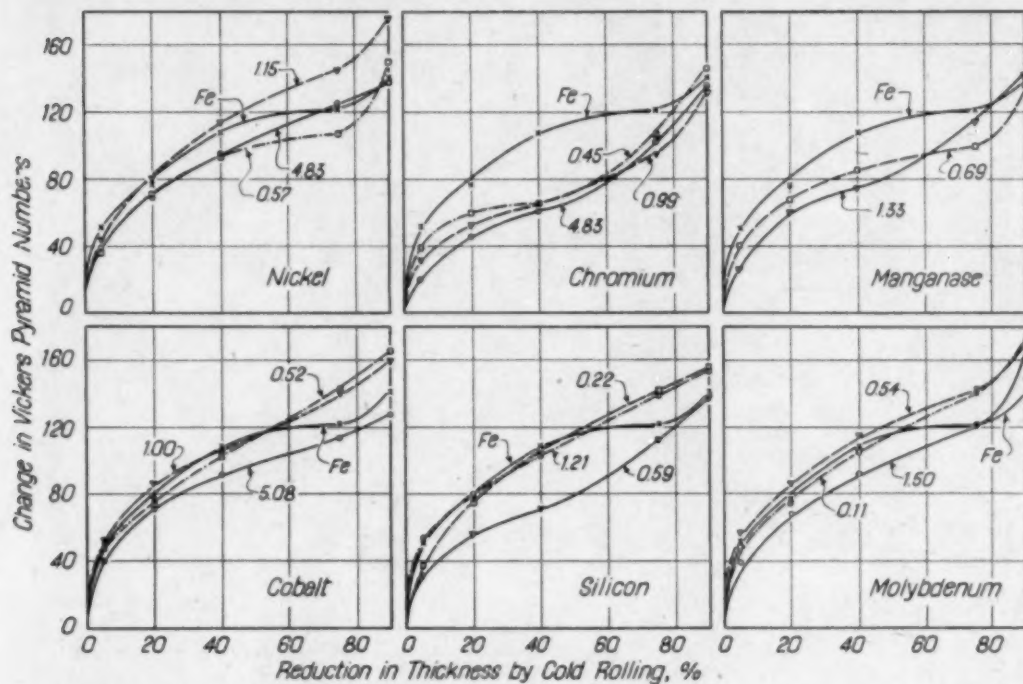


Fig. 12—The Relation Between Degree of Cold Deformation as Measured by Reduction of Thickness on Rolling and the Vickers Pyramid Hardness for the Various Binary Alloys.

STUDIES ON THE BINARY ALLOYS

The progress of work hardening of the alloys as a consequence of cold rolling is shown in Fig. 12 for each alloy by plotting change in Vickers hardness as a function of percentage reduction in thickness. For purposes of comparison, the curve for unalloyed iron is plotted on each graph. In most cases, the curves for the alloys are practically superimposed upon that for the unalloyed iron; the notable exceptions being the chromium alloys, the manganese alloys, and isolated cases in the silicon, cobalt, and molybdenum alloys. It is obvious that if the actual hardness values were plotted against per cent reduction in thickness, all curves would lie above the curve for unalloyed iron as a consequence of the hardening by solid solution as demonstrated for these alloys by Austin (15). In addition to the positions of the curves for the alloys relative to that for the unalloyed iron, the other noticeable differences are the elimination of the flat spot evident between 40 and 75 per cent reduction on the alloy curves, as compared to the curve for iron and the absence of

the definite upward swing of the end of the curve in the case of many of the alloys. Examination of the alloys in the as-rolled condition showed no differences in microstructure as compared to unalloyed iron with the exceptions of grain size variations.

The changes in hardness of the alloys as a function of annealing temperature are shown in Figs. 13 to 15. These diagrams show

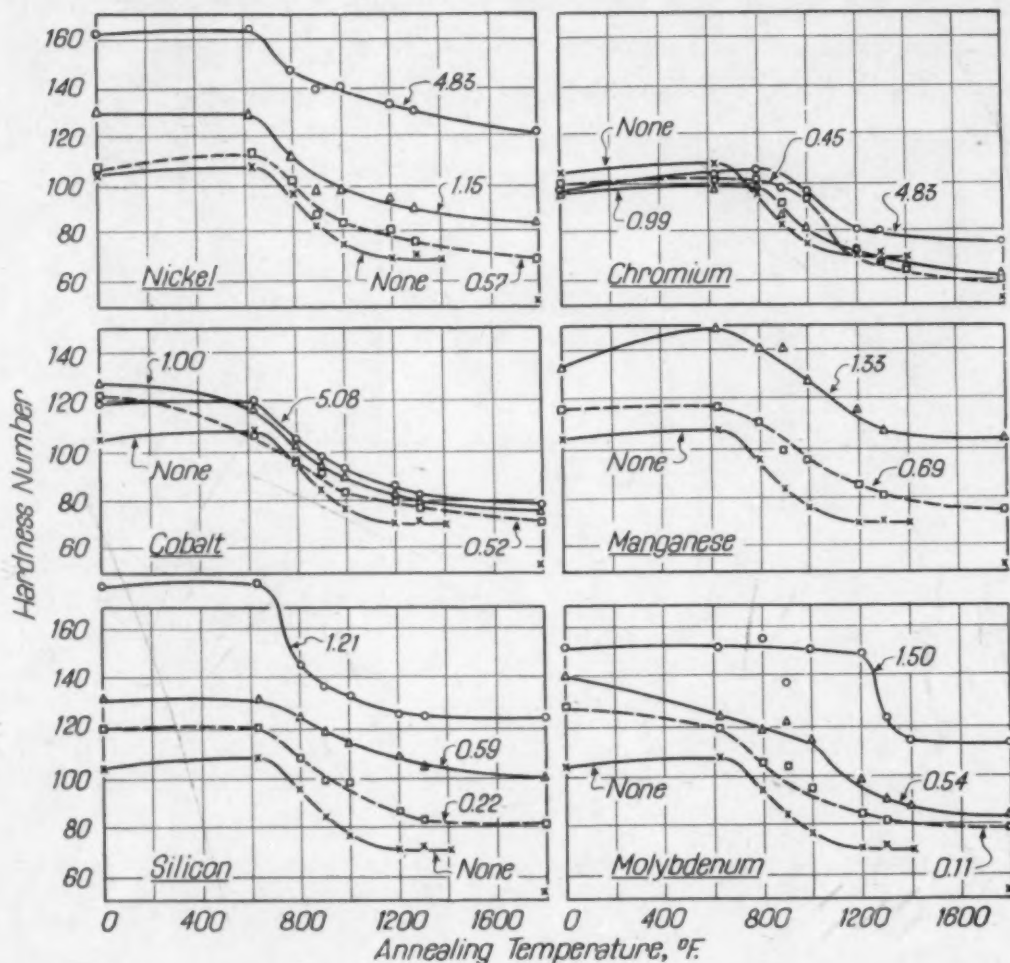


Fig. 13—Relation Between Annealing Temperature and Vickers Pyramid Hardness for the Various Binary Alloys Reduced 5 Per Cent in Thickness by Cold Rolling.

only the results for the samples reduced 5 per cent, 40 per cent, and 90 per cent in thickness. The annealing curves for unalloyed iron have been included on the respective graphs. The curves for the other reductions are quite similar and show a gradual transition with increasing amounts of cold rolling from the type of curve depicted in Fig. 13 to that drawn in Fig. 15.

The effect of time upon the softening of the alloys at 480 degrees Cent. (900 degrees Fahr.) was studied in order that a direct

comparison could be made with the behavior of pure iron, presented earlier in the paper. The complete data have been presented in the form of bar graphs, reproduced as Fig. 16. The numbers at the base of the bars are indicative of the annealing time in hours. The letters "A" and "O" indicate the annealed and as-rolled specimens respectively. The total height of any bar from the base of the diagram to the top of that particular bar represents the hardness for the alloy under the indicated conditions of prior reduction and annealing time at 480 degrees Cent. (900 degrees Fahr.).

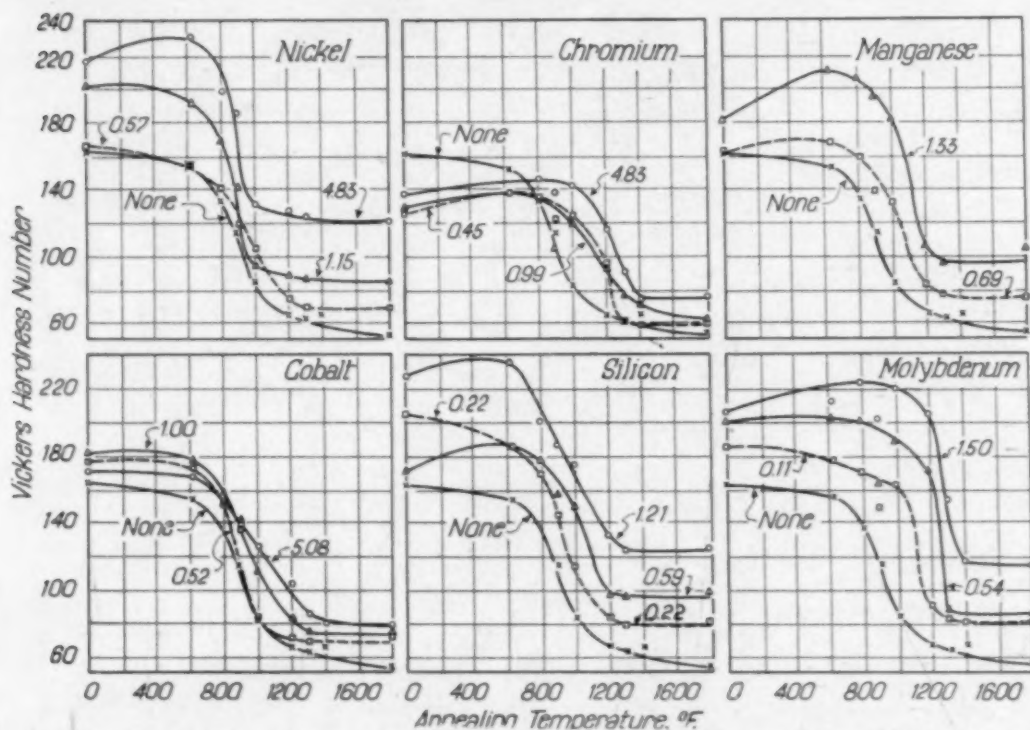


Fig. 14—Relation Between Annealing Temperatures and Vickers Pyramid Hardness for the Various Binary Alloys Reduced 40 Per Cent in Thickness by Cold Rolling.

DISCUSSION OF RESULTS

In reviewing the literature on this subject, it seemed to the authors that a great deal of confusion exists in the terminology regarding the annealing of cold-worked metals and alloys. Accordingly, a few comments seem desirable before discussing the results of the investigation.

Various processes are considered to be involved in the temperature softening curves of cold-worked metals and alloys. Recovery, recrystallization, and grain growth occur dependent upon the temperature and time of anneal with relation to the prior history of the material concerned. A further general conclusion seems to be that

these processes may be sharply defined in the case of some metals and they may overlap one another in other instances.

The following definitions seem to be held rather generally:

Recovery—A change in the properties of cold-worked metals in the direction of the state existing prior to deformation. These changes are not accompanied by any change in the microstructure.

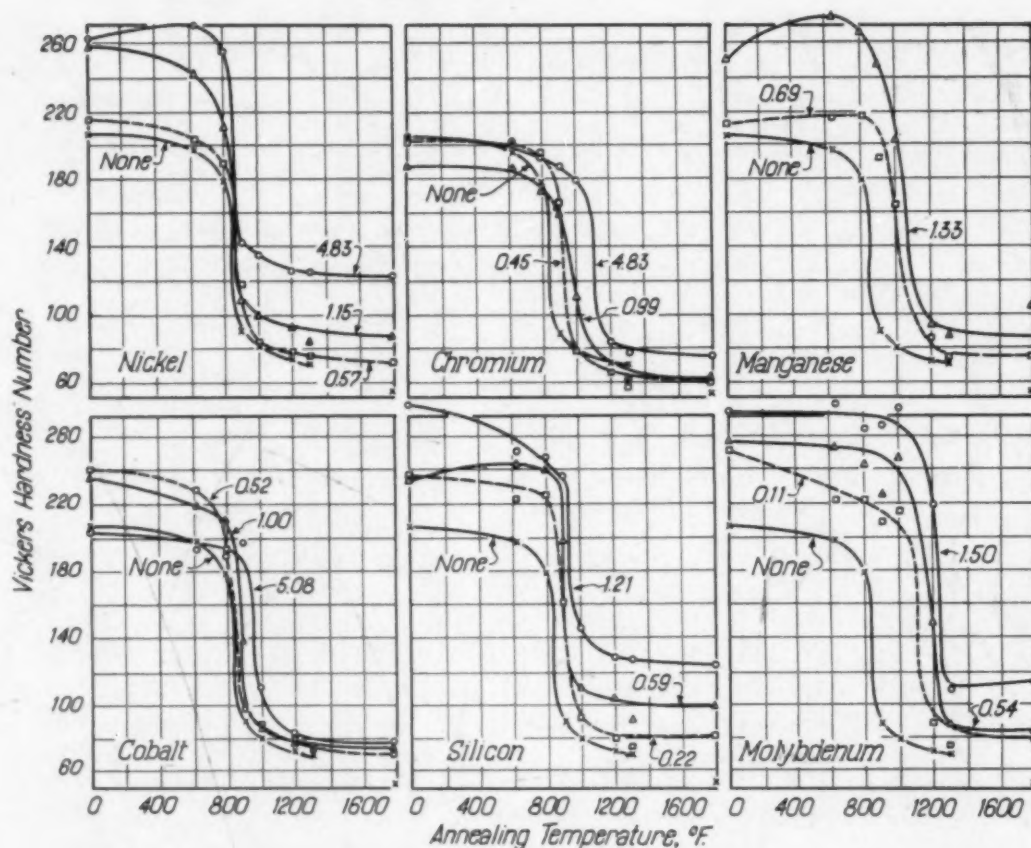


Fig. 15—Relation Between Annealing Temperatures and Vickers Pyramid Hardness for the Various Binary Alloys Reduced 90 Per Cent in Thickness by Cold Rolling.

Recrystallization—The formation and growth of new strain-free grains by heating metals and alloys that have been cold-worked. With complete recrystallization the unstrained grains supplant entirely the cold-worked structure. Due to the pronounced changes in structure, the microscope affords a convenient means of following recrystallization. The changes may be followed also quite accurately by X-ray studies. Major changes in the mechanical properties occur coincidental with recrystallization and the process may or may not be followed accurately by following such changes.

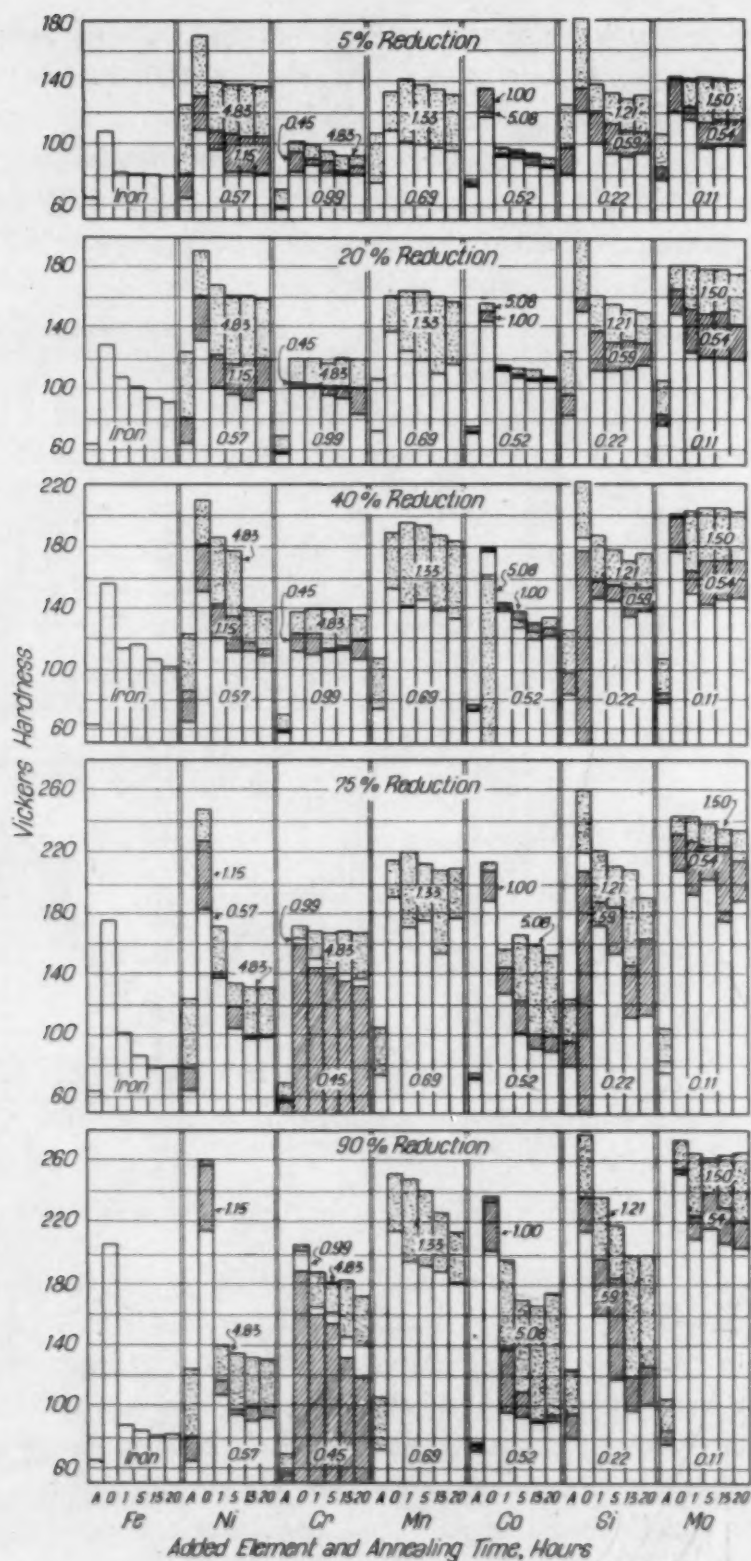


Fig. 16—Effect of Time of Annealing at 900 Degrees Fahr. on the Vickers Pyramid Hardness of the Commercial Iron and of all the Binary Alloys After Reducing 5, 20, 40, 75 and 90 Per Cent by Cold Rolling. Designations "A" and "O" Refer to Dead Soft Annealed and Cold-Rolled States Respectively. The Numbers 1 to 20 Relate to Time of Annealing.

Grain Growth—This term, as generally used, refers to the growth of the strain-free grains during and after recrystallization. It has been recognized, however, that grain growth may occur, without recrystallization, by the simple mechanism of boundary migration. Carpenter (16) many years ago presented experimental evidence of this phenomenon.

THE DEFORMATION AND ANNEALING OF UNALLOYED IRON

The progressive change of hardness of the unalloyed iron during cold rolling and of the microstructural appearance of the rolled structures (Figs. 1 and 2) is common to metals and alloys, and requires no specific comment. Furthermore, the results obtained during the annealing of samples reduced 40, 75, and 90 per cent in thickness are perfectly straightforward and changes in hardness during annealing can be correlated quite well with changes in microstructure.

The softening at the lower annealing temperatures appears to be the consequence of recovery since no changes in microstructure were observed after annealing at 330 degrees Cent. (625 degrees Fahr.) (for each of the three reductions) and after annealing at 425 degrees Cent. (800 degrees Fahr.) for 40 per cent reduction.

Definite evidences of recrystallization can be noted at 425 degrees Cent. (800 degrees Fahr.) for samples reduced 75 and 90 per cent in thickness (Figs. 6, 7 and 8) and at 480 degrees Cent. (900 degrees Fahr.) for the samples reduced 40 per cent in thickness. In all three samples a sharp change in direction in the hardness curves was indicative of the fact that with a small increase in temperature total resistance to any recrystallization was followed by rapid softening and by complete recrystallization. This temperature interval was a minimum with the sample reduced 90 per cent and extended over the greatest temperature interval with 40 per cent reduction. Some grain growth occurred with all three reductions. The recrystallized grain was quite fine with the samples reduced 75 and 90 per cent and consequently grain growth was quite noticeable. The sample reduced by 40 per cent in thickness showed medium grain size on recrystallization and grain growth was slight above the recrystallization temperature.

The situation is not quite so clear with the samples reduced 5 and 20 per cent in thickness. There is reasonably good evidence

with the sample reduced 20 per cent that recrystallization initially occurs at about 540 degrees Cent. (1000 degrees Fahr.). Softening at temperatures below 540 degrees Cent. (1000 degrees Fahr.) seems to be caused wholly by recovery. When recrystallization does occur at 540 degrees Cent. (1000 degrees Fahr.) and above, the recrystallized grain is large and does not show much further growth, even at a temperature of 870 degrees Cent. (1600 degrees Fahr.). This is apparently dependent on limited nucleation with low reduction per cent.

These results are summarized in the three-dimensional diagram of Fig. 9. The following facts are apparent from this diagram:

(a) As the amount of reduction is increased from 20 to 90 per cent, the temperature of the beginning of recrystallization is lowered from 540 to 425 degrees Cent. (1000 to 800 degrees Fahr.).

(b) The grain size just after recrystallization is complete is finer as the percentage reduction becomes greater.

(c) Recrystallization occurs over a sharply limited temperature interval with high reductions and a less well defined and broader temperature interval with lower reductions.

(d) Grain growth of the recrystallized grains is most pronounced where the recrystallized grains were fine (75 and 90 per cent reductions) and is less evident when the grains were coarser just after recrystallization (20 and 40 per cent reductions).

The behavior of the sample reduced 5 per cent in thickness requires separate comment. No change in the microstructure was noted until the annealing temperature attained 870 degrees Cent. (1600 degrees Fahr.) and then exceptionally large grains were developed. Hence, the softening at temperatures lower than this was caused apparently by recovery. It is probable that the grain coarsening at 870 degrees Cent. (1600 degrees Fahr.) resulted without true recrystallization and was dependent on grain boundary migration.

The effect of time upon the softening of cold-rolled iron was studied at 480 degrees Cent. (900 degrees Fahr.). The reason for the choice of this particular temperature was stated previously. It was also pointed out in reference to Fig. 10 that, for any given annealing time, there was a maximum in the hardness-per cent reduction curves for each of the annealing temperatures studied and this maximum was associated with the samples reduced 40 per cent

in thickness. This observation would seem to be dependent on the differences in the causes of softening. The samples reduced 5 and 20 per cent apparently soften by recovery. No change was noted in the microstructure up to 20 hours annealing time. On the other hand, samples reduced in thickness 40, 75, and 90 per cent softened mainly by recrystallization. Recrystallization is rapid and quite complete with the heavier reductions, but is incomplete even after 20 hours in the sample reduced 40 per cent. This would mean that some strain-hardened regions still exist in the sample and probably contribute to keeping the hardness up to a slightly higher level, with this reduction, as compared to the other samples. This is confirmed by an examination of the photomicrographs in Fig. 11.

THE DEFORMATION AND ANNEALING OF THE ALLOYS

The curves relating changes of hardness to per cent reduction of thickness expressed as $\frac{T_o - T_r}{T_o} \times 100$ were given in Fig. 12, where " T_o " is the original thickness prior to rolling and " T_r " is the thickness at any stage of reduction. Some of these curves have been re-plotted in Fig. 17 to show the relation between changes of hardness and reduction in thickness as expressed by the quantity: $2.303 \log_{10} \frac{T_o}{T_r}$. This is in a sense an expression of true deformation or true reduction of thickness. Cook and Richards (17) have made similar plots in studying the structural changes effected in 70:30 brass strip. They believe that sharp inflections of the type found between 75 and 90 per cent reduction in thickness in curves plotted as in Fig. 12 have no particular significance, but are due to the fact that the percentage reduction of thickness as expressed in this figure is not a proportional measure of deformation. They feel that when reduction in thickness is expressed in logarithmic fashion as in Fig. 17 a true proportional scale is provided for deformation. When this is done the latter portion of the curves at higher deformations assumes an almost linear course, although it will be noted from Fig. 17 that some of the curves still exhibit a slight turn upward between the two highest amounts of cold deformation. In their consideration of brass Cook and Richards point out that such curves may be divided into two portions, the first extending up to approximately 40 per cent and the second covering the range of higher reductions. Their microstructural results indicate that the first part of the curve

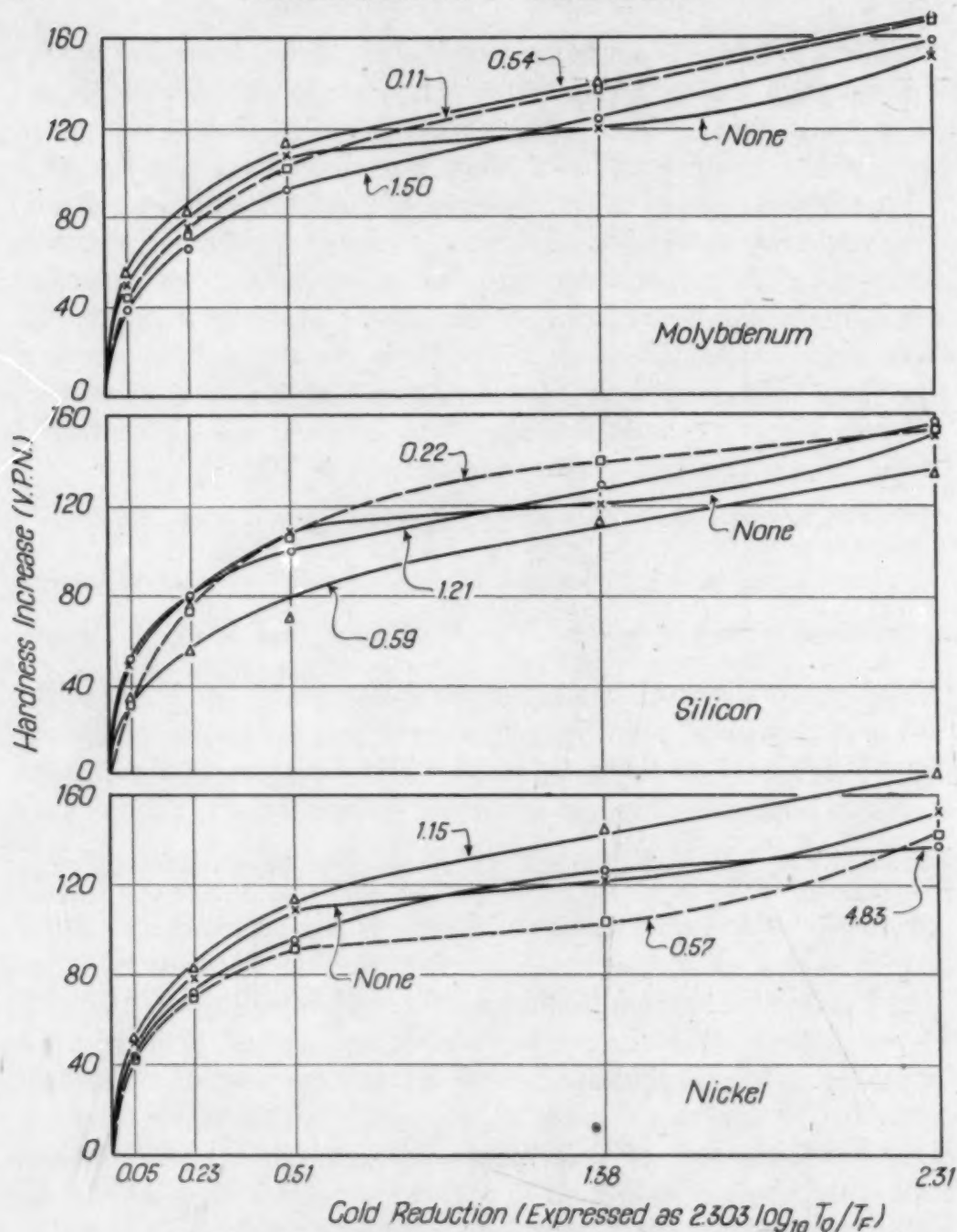


Fig. 17—Relation Between Degree of Cold Deformation Expressed as $2.303 \log_{10} \frac{T_0}{T_f}$ and the Vickers Pyramid Hardness for Molybdenum, Silicon, and Nickel. T_0 is Original Thickness and T_f the Thickness At Any Given State of Cold Reduction.

is associated with crystal slip and the second part with crystal breakup.

The annealing curves for the alloys are very similar in shape to those for unalloyed iron. The complete data used in plotting these curves are presented in Tables IV to VI. In order to compare the

Table IV
Effect of Annealing for 1 Hour at the Temperatures Indicated, on the Vickers Pyramid Hardness, After Cold Rolling the Various Alloys
5 and 20 Per Cent Reduction in Thickness

Alloy	Per Cent	5 Per Cent Reduction in Thickness							20 Per Cent Reduction in Thickness							As Rolled		
		As Rolled	625	800	900	1000	1200	1300	1800°F	53	130	625	800	900	1000	1200	1300	1800°F
None	105	109	97	83	77	71	72	53	130	123	114	107	104	64	61	53	
Nickel . . .	{ 0.57	107	114	103	87	85	84	78	71	142	138	123	102	99	88	70	71	
	{ 1.15	131	130	113	199	100	96	93	87	167	153	138	122	124	88	82	87	
	{ 4.83	163	165	148	140	141	134	132	123	192	197	178	168	128	121	118	123	
Chromium . . .	{ 0.45	99	100	102	91	95	69	68	60	120	119	122	103	112	83	78	60	
	{ 0.49	95	97	99	87	82	72	71	63	117	121	123	98	106	90	82	63	
	{ 4.83	97	103	106	98	96	81	81	76	122	121	119	118	118	106	102	76	
Manganese . . .	{ 0.69	117	118	113	100	97	86	81	76	144	144	133	125	114	96	76	76	
	{ 1.33	133	150	142	142	129	117	108	107	167	182	181	164	167	113	97	107	
Cobalt . . .	{ 0.52	121	107	98	91	84	81	77	72	145	135	129	113	104	78	71	72	
	{ 1.00	128	118	102	92	90	85	82	76	168	149	125	114	110	92	76	71	
	{ 5.08	120	120	105	98	94	87	81	79	151	143	133	114	116	105	97	79	
Silicon . . .	{ 0.22	120	121	107	98	99	86	83	83	159	160	138	113	110	85	78	83	
	{ 0.59	133	133	126	119	115	109	106	100	157	163	152	137	138	95	92	100	
	{ 1.21	179	180	147	137	134	126	127	125	205	199	177	161	158	142	137	125	
Molybdenum . . .	{ 0.11	129	120	106	105	96	85	82	80	155	159	137	124	121	107	80	80	
	{ 0.54	142	125	119	123	115	99	90	85	170	162	160	153	141	123	106	85	
	{ 1.50	153	153	157	138	151	151	124	114	182	192	188	181	186	178	146	114	

Table V
Effect of Annealing for 1 Hour at the Temperatures Indicated, on the Vickers Pyramid Hardness, After Cold Rolling the Various Alloys
40 and 75 Per Cent Reduction in Thickness

Alloy	Per Cent	40 Per Cent Reduction in Thickness										75 Per Cent Reduction in Thickness									
		As Rolled	625	800	900	1000	1200	1300	1800°F.	As Rolled	625	800	900	1000	1200	1300	1800°F.				
None	162	153	134	114	83	65	63	53	175	161	157	101	76	71	69	53				
Nickel . . .	{ 0.57	165	151	141	119	106	74	69	71	178	181	160	142	85	82	76	71				
	{ 1.15	202	193	169	142	95	90	88	87	233	205	199	138	99	92	87	87				
	{ 4.83	216	232	199	186	130	128	125	123	249	249	242	172	133	131	131	123				
Chromium . . .	{ 0.45	126	139	133	122	124	96	60	60	167	154	158	151	121	65	64	60				
	{ 0.99	129	138	136	105	120	91	77	63	159	156	150	144	138	72	68	63				
	{ 4.83	138	143	146	138	142	116	91	76	180	175	167	168	164	82	77	76				
Manganese . . .	{ 0.69	162	169	161	140	133	83	77	76	176	197	186	172	157	83	81	76				
	{ 1.33	182	212	207	196	183	107	86	107	222	236	233	221	202	114	101	107				
Cobalt . . .	{ 0.52	177	173	151	136	86	72	69	72	215	183	173	127	84	80	77	72				
	{ 1.00	182	178	158	142	111	84	75	76	218	199	183	157	93	86	78	76				
	{ 5.08	171	168	152	138	126	104	86	79	193	185	178	146	149	89	84	79				
Silicon . . .	{ 0.22	192	186	170	145	115	85	80	83	225	215	197	172	99	90	88	83				
	{ 0.59	171	186	178	157	150	99	98	100	213	218	210	189	113	109	106	100				
	{ 1.21	228	236	201	187	176	133	124	125	264	262	240	222	162	136	133	125				
Molybdenum . . .	{ 0.11	186	177	170	148	162	90	81	80	222	203	197	192	197	92	85	80				
	{ 0.54	201	203	202	163	188	171	87	85	227	227	238	228	224	181	99	85				
	{ 1.50	207	213	224	202	219	205	152	114	236	242	247	245	251	213	120	114				

effects of the added elements on the resistance to softening of iron, it is necessary to decide upon some basis for comparison. The softening temperature chosen here was the half hard point, that is the temperature at which the hardness of the annealed alloy lies midway between that of the rolled and the fully annealed metal. This selected condition is that at which the rate of change in hardness as a function of temperature of anneal is a maximum (the point of inflection of the curve) and hence is the condition most easily and

Table VI
Effect of Annealing for 1 Hour at the Temperatures Indicated, on the Vickers Pyramid Hardness, After Cold Rolling the Various Alloys 90 Per Cent Reduction in Thickness

Alloy	Per Cent	90 Per Cent Reduction in Thickness							
		As Rolled	625 °F.	800 °F.	900 °F.	1000 °F.	1200 °F.	1300 °F.	1800 °F
Pure Iron	206	197	178	89	80	73	71	53
Nickel	{ 0.57	215	203	189	117	82	77	75	71
	{ 1.15	258	242	210	108	99	92	84	87
	{ 4.83	262	272	256	141	134	125	124	123
Chromium	{ 0.45	203	201	197	166	78	67	54	60
	{ 0.99	188	187	173	160	111	71	62	63
	{ 4.83	206	202	194	188	180	84	77	76
Manganese	{ 0.70	213	216	218	193	165	86	72	76
	{ 1.30	251	278	268	249	203	93	87	107
Cobalt	{ 0.52	238	228	192	97	89	79	76	72
	{ 1.00	235	219	210	138	87	80	74	76
	{ 5.08	203	192	188	197	111	84	75	79
Silicon	{ 0.22	236	222	225	160	92	80	74	83
	{ 0.59	235	243	240	197	110	104	92	100
	{ 1.21	279	251	247	236	146	129	128	125
Molybdenum	{ 0.11	251	221	222	209	215	90	75	80
	{ 0.54	255	253	243	225	247	150	87	85
	{ 1.50	274	279	264	266	276	219	109	114

precisely defined. Such softening temperatures were noted from the respective annealing curves and are listed in Table VII.

It is apparent from this table that molybdenum, chromium, and manganese are quite effective in raising the softening temperature of iron with all deformations. A more specific comparison can be made at approximately 1 weight per cent of added element. It happens that this also affords a comparison at 1 atomic per cent of added element. The following remarks will then apply:

Molybdenum is extremely effective in raising the softening temperature of iron, the increase with all reductions being of the order of 95 to 205 degrees Cent. (200 to 400 degrees Fahr.). This element is followed in effectiveness by chromium and manganese which are quite similar in behavior and raise the softening temperature by

about 95 to 150 degrees Cent. (200 to 300 degrees Fahr.). Manganese seems to be relatively ineffective with samples reduced 5 and 20 per cent in thickness. In raising the softening temperature by 80 to 100 degrees Fahr. silicon is only mildly effective, particularly with the lower percentages of reduction. Cobalt and nickel do not have any marked effect on raising the temperature of softening with any of the various percentages reductions.

Fig. 18 is included here to portray graphically the behavior of

Table VII
Softening Temperatures of Commercially Pure Iron and of the Various Binary Alloys
After Cold Reduction by Rolling 5, 20, 40, 75 and 90 Per Cent in Thickness

Alloy	Per Cent	Reduction in Thickness, Per Cent				
		5	20	40	75	90
		Softening Temperature, °F.				
None	870	1070	900	880	830
Nickel	{ 0.57	960	980	940	890	860
	{ 1.15	860	960	880	900	860
	{ 4.83	940	860	900	910	860
Chromium	{ 0.45	1100	1140	1200	1010	940
	{ 0.99	1050	1200	1180	1100	980
	{ 4.83	1120	1300	1240	1120	1100
Manganese	{ 0.69	1000	1040	1060	1080	1000
	{ 1.33	1100	1100	1120	1100	1040
Cobalt	{ 0.52	800	980	880	880	860
	{ 1.00	800	920	940	900	890
	{ 5.08	800	1050	1000	1040	960
Silicon	{ 0.22	900	900	900	920	900
	{ 0.59	980	1020	1060	940	900
	{ 1.21	760	930	980	960	940
Molybdenum	{ 0.11	820	1020	1120	1100	1100
	{ 0.54	1020	1140	1240	1220	1180
	{ 1.50	1280	1300	1290	1240	1220

the different elements investigated. This presentation is for approximately 0.5 weight per cent of added element and shows the relative potency of each element in affecting the softening temperature of cold-rolled iron for each reduction percentage used. It is interesting to note for 20 per cent reduction that many of the elements lowered the softening temperature slightly and even chromium and molybdenum were only mildly effective in raising this temperature.

In the majority of the added elements the lowest weight per cent added caused the greatest increase in softening temperature. Increasing the amount beyond this had little further effect.

The results are in general agreement with those reported by Tammann (refer to Table I). The mechanism by which these elements affect the softening temperature is not clear. It is interesting

to note from the results of this work and further unpublished research on this subject, as well as from Tammann's work, that elements with the same crystal structure at room temperature as iron seem to be the ones effective in raising the softening temperature—molybdenum, chromium, vanadium, and tungsten are all effective. Manganese, with a complex cubic structure, is also effective. There

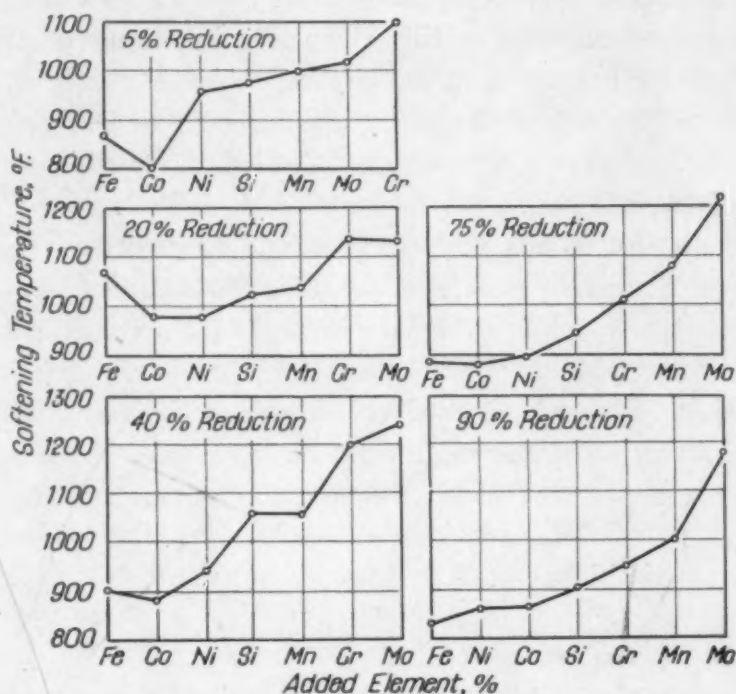


Fig. 18—Relative Effects of 0.5 Per Cent (Approx.) of the Various Elements in Solid Solution in Iron on Raising the Softening Temperature After Cold Rolling 5, 20, 40, 75 and 90 Per Cent Reduction in Thickness. Softening Temperature Has Been Defined as That Temperature of Annealing for a Period of 1 Hour at Which the Hardness of the Alloy Lies Midway Between That of the Rolled and Fully Annealed Alloy.

is some evidence that this observation of crystallographic similarity holds true with nonferrous metals, e. g., nickel and silver raise the softening temperature of copper (18), (19) and silver raises the softening temperature of lead (20).

The effects of the various elements on solid solution hardening do not seem to bear any direct relation to the effects of the same elements on softening temperature. An opinion (21) has been expressed that elements causing marked solid solution hardening, probably as a consequence of lattice distortion, might be expected to oppose the removal of any additional lattice distortion such as that caused by cold work, for example. This does not agree with the

present findings. One may compare the behavior of chromium and molybdenum, and cobalt and nickel in this respect. Molybdenum and nickel have been shown to produce a considerable amount of hardening by solid solution in iron, quite probably by lattice distortion. Molybdenum raises the softening temperature markedly while nickel is ineffective. Chromium and cobalt do not produce much hardening as a consequence of their solid solubility in iron, but chromium is quite effective in raising the softening temperature.

The bar graphs in Fig. 16 were included to show the relative effects of the added elements on the rate of softening at 900 degrees Fahr. The results on unalloyed iron at this temperature have been included to give a basis of comparison. In general the behavior was quite in line with what would be expected from a consideration of Figs. 13 to 15. The alloys containing molybdenum, manganese, or chromium were quite resistant to softening at this temperature for any per cent reduction, with molybdenum being most effective. The silicon and cobalt alloys showed some retardation of softening with the higher percentages of reduction, but the elements were ineffective with lesser percentages of reduction (5 and 20 per cent). Nickel was ineffective in opposing softening with all reductions of thickness.

CONCLUSIONS

(A) Most of the binary alloys were similar to unalloyed iron in their strain hardening characteristics. However, some alloys did show deviations in such manner that with lower amounts of reduction, strain hardening of the alloys was not so rapid as in unalloyed iron. Certain inflections in the curves showing change of hardness with increase in reduction of thickness were eliminated by plotting deformation as a logarithmic function of initial and final thicknesses.

(B) Annealing studies were conducted on unalloyed iron reduced 5, 20, 40, 75, and 90 per cent in thickness. It was found that softening at the lower temperatures occurred as a consequence of recovery. Annealing at higher temperatures caused recrystallization with all degrees of deformation excepting the 5 per cent reduction. The latter sample softened at all temperatures by recovery and exhibited marked grain growth at 1600 degrees Fahr. (870 degrees Cent.), possibly as a consequence of boundary migration without recrystallization.

(C) The following points were noted from the instances in which recrystallization occurred:

(a) The temperature of beginning of recrystallization was lowered as the degree of deformation was increased. Thus, recrystallization was initiated at 1000 degrees Fahr. (540 degrees Cent.) with samples reduced 20 per cent in thickness, while with samples reduced 90 per cent in thickness the temperature was lowered to 800 degrees Fahr.

(b) Grain size immediately after recrystallization was finer as degree of deformation was increased.

(c) Growth of the grains developed by recrystallization was most pronounced with fine recrystallized grains.

(D) The softening curves for the cold-worked binary alloys were quite similar in contour to those for unalloyed iron. However, the softening temperature, defined as the mid-point between the as-worked hardness and the fully annealed hardness, was raised by certain of the alloying elements. Molybdenum was most potent in this respect raising this temperature by as much as 400 degrees Fahr. (205 degrees Cent.) in some instances. Chromium and manganese were also quite effective, while silicon was only mildly so. Cobalt and nickel had no effect in comparison with the unalloyed iron. It was noted that the softening temperature was raised most pronouncedly by the initial small addition of the alloying element and only slightly more so by larger additions.

(E) The effect of time as a variable in the annealing process was studied at 900 degrees Fahr. (480 degrees Cent.), at which temperature softening occurred very definitely and yet was not too rapid to be followed conveniently. It was found with unalloyed iron that softening occurred by recovery in samples reduced 5 and 20 per cent and by practically complete recrystallization in samples reduced 75 and 90 per cent. Recrystallization was only partially complete in the sample reduced 40 per cent and this sample did not soften to the same extent as the others. In all cases, hardness decreased rapidly in the first hour and more slowly thereafter.

(F) Similar studies were made at 900 degrees Fahr. (480 degrees Cent.) with the binary alloys. Molybdenum, manganese, and chromium retarded softening at this temperature as compared to unalloyed iron, with molybdenum being the most effective. Silicon and cobalt caused a moderate decrease in rapidity of softening with severely deformed samples, but exerted no effect with lower reductions. Nickel was relatively ineffective in all instances.

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DISCUSSION

Written Discussion: By F. R. Morral, metallurgist, Metal Trades Laboratory, American Cyanamid Co., Stamford, Conn.

The authors of this paper are to be congratulated because they have continued their interesting work on pure binary iron alloys. This type of investigation seems to be necessary to secure data to establish the correctness of "guesses," based on the behavior of ordinary steels, which have been made in the past; and to further add to the knowledge of iron and its alloys.

A curve has been drawn apparently rather arbitrarily through some points in Fig. 1. The authors claim that around 40 per cent reduction the curve tends to become parallel to the horizontal. In view of the data presented, this conclusion can be disputed. The authors need data, say at 50 per cent reduction, to really establish the contour of the curve. One might expect some similarity with the curves shown in Fig. 12, page 464, which are nearly parallel. Only a few points in certain instances need checking.

A point in Fig. 3 appears to be out of place, namely, for 20 per cent reduction, annealed at 1000 degrees Fahr. Was this particular experiment repeated for check purposes? All other similar curves shown in Figs. 13 to 15 run nearly parallel.

Some of the remarks on pages 475 to 477 as to the effect of the various alloying elements on the softening temperature are too general and therefore misleading. For example, according to the data presented in Table VII, in the case of 0.11 per cent weight of molybdenum, a lowering of the softening temperature was found. Also, silicon with 20 per cent reduction at all compositions showed a lowering.

In Table A a comparison is made using the published data on the increase of the softening temperature by alloying elements after 90 per cent reduction by cold rolling. The degree of agreement between Tammann's work and the present is good in certain cases and not so good in others.

Table A
Increase in Softening Temperature (in Degrees Fahr.) with Various Percentages of Alloying Elements

Alloying Element Per Cent Atomic (Approx.)	Austin's Data				Tammann's Data (p. 449)			Crystal Structure	Distance* of Closest Approach A	Co-or- dina- tion No.
	0.5	1.0	2	4.0	1	2	4			
Iron	b.c.c.	2.4777	8
Aluminum	175	f.c.c.	2.8577	12
Nickel	30° F.	30	30	30	175	f.c.c.	2.4868	12
Cobalt	30	60	95	130	250	h.c.p.	2.499	12
Silicon	70	100	155	176	...	diamond	2.3457	4
Chromium	115	150	230	350	b.c.c.	2.4929	8
Manganese	135	185	240	f.c.tetr.	2.582	8
Vanadium	212	...	b.c.c.	2.6274	8
Molybdenum	350	380	405	450	...	b.c.c.	2.7196	8
Tungsten	464	b.c.c.	2.7354	8

*Barrett, Structure of Metals, 1942, p. 552-4.

It appears from those data that crystal structure, as pointed out by the authors, is not the only factor to be considered to explain the raise in the

softening temperature. Closest approach of atoms (or atomic radii) may be more effective. The elements are listed in the table in the order of increasing the softening temperature of iron. The distance of closest approach of the atoms in Å of the effective b.c.c. elements show a definite trend. It may also be noted that the elements that have not affected the softening temperature have a crystal structure with a co-ordination number of 12, while those which have affected the softening, irrespective of crystal structure, have co-ordination numbers of 8 and 4, respectively, with the exception of cobalt.

All work seeking fundamental metallurgical information as done so notably by the authors should be encouraged.

Written Discussion: By G. M. Butler, associate director of research, Allegheny Ludlum Steel Corp., Dunkirk, N. Y.

The results presented in this very interesting paper substantiate and augment those obtained in a somewhat similar study cited as reference No. 3. In this earlier work, the recovery and recrystallization characteristics of some binary alloys of iron with nickel, chromium, and molybdenum were compared with those of "Wemco" iron. The alloys were melted under vacuum obtainable from a Megavac pump and cold-drawn into wires 0.04-inch round from a series of initial sizes to give varying degrees of cold reduction. After treating pieces of the wires at various temperatures, their hardness, tensile strength, and electrical resistance were measured and compared with microstructural changes.

Softening temperatures representing inflection points on the hardness-temperature curve, comparable to data in the authors' Table VII, were as follows:

Alloy	Per Cent	Softening Temperature, Degrees Fahr.	
		40 Per Cent Reduction in Section	98 Per Cent Reduction in Section
None	...	1000	915
Nickel	0.24	1000	970
	2.07	1020	970
Chromium	0.10	1040	970
	1.72	1110	1020
Molybdenum	0.27	1190	1060
	1.82	1360	1240

Other percentage reductions and intermediate analyses were studied, but the trend of these is clearly shown in the present paper.

Incipient recrystallization appeared under the microscope in "Wemco" iron at temperatures as low as 790 degrees Fahr. (420 degrees Cent.) when cold-drawn 98 per cent; at 840 degrees Fahr. (450 degrees Cent.) after 90 per cent reduction; and 930 degrees Fahr. (500 degrees Cent.) after 77 per cent reduction. In all the chromium and nickel alloys, with up to 2 per cent of alloy, wires cold-drawn to 98 per cent reduction in area all seemed to show new grains at about 850 degrees Fahr. (455 degrees Cent.). In the similar wires of the molybdenum alloys, however, this appearance of recrystallization was elevated from 1000 degrees Fahr. (540 degrees Cent.) in the 0.25 per cent molybdenum wire to 1110 degrees Fahr. in the 2 per cent molybdenum wire.

Lesser degrees of cold work were more helpful in differentiating between the recrystallization tendencies of the various alloys. The greatest differences appeared when the reduction of area by drawing was about 30 to 40 per cent. At this degree of reduction, recrystallization first appeared at 1020 degrees Fahr. (550 degrees Cent.) with 0.10 per cent chromium, 1110 degrees Fahr.

(600 degrees Cent.) with 1.7 per cent chromium, 1000 degrees Fahr. (540 degrees Cent.) with 0.25 per cent nickel, 1040 degrees Fahr. (560 degrees Cent.) with 2 per cent nickel, 1200 degrees Fahr. (650 degrees Cent.) with 0.25 per cent molybdenum, and 1390 degrees Fahr. (755 degrees Cent.) with 2 per cent molybdenum.

Time-hardness studies made at 180-degree Fahr. intervals on wires cold-drawn to 98 per cent reduction in area showed only a slight softening at 750 degrees Fahr. (400 degrees Cent.) in any of the wires in the first few minutes, with no subsequent decrease in periods up to 2 hours.

At 930 degrees Fahr. (500 degrees Cent.) the "Wemco" iron softened quite rapidly at first but reached a constant value after about 45 minutes. The nickel and chromium alloys softened at this temperature more gradually and over a longer period. At the end of 2 hours, all the nickel alloys had completely softened, while the 0.10 per cent chromium alloy had still a little farther to go and the 1.7 per cent chromium alloy had only dropped half-way down to its minimum softness. The molybdenum alloys showed no softening at this temperature in times up to 4 hours.

At 1110 degrees Fahr. (600 degrees Cent.), all the alloys except 2 per cent molybdenum softened completely in 10 minutes. The 2 per cent molybdenum wire had not started to soften in 2 hours. No compositions between 0.25 per cent and 2 per cent molybdenum were studied in this series.

Tensile tests, including stress-strain curves, were made on many of the wires after treating at various temperatures. Generally the yield points and breaking loads fell off with increasing temperature in very nearly the same manner as the hardness values. Stress-strain curves on the as-drawn wires lacked any linear portion in the elastic range, but treating as low as 390 degrees Fahr. (200 degrees Cent.) was sufficient to restore linearity in the elastic range.

Wires which had largely recrystallized showed much lower breaking loads and sharp yield points. It did not appear, however, that tensile tests were sufficiently sensitive to incipient recrystallization to be of value in studying the early stages of new crystal formation.

Electrical resistance measurements were also used to determine changes in the various wires after treating at increasing temperatures. It became apparent that the resistance of the cold-drawn wires decreased materially at temperatures far below those at which the hardness values started to drop. Not the slightest evidence of the recrystallization range could be seen in the resistance curves. An unexpected increase in resistance of many of the wires after treating at 1475 degrees Fahr. (800 degrees Cent.) was found; but this may have been due to some kind of contamination during treating.

Resistance, like hardness and strength, increased at first rapidly in the intermediate ranges of working, then again accelerated as the most drastic degrees of working were approached. The shape of the curves in Fig. 12 of the present paper is entirely representative of those for all the properties studied in the work cited. The flat spot shown by the present authors on the curve for pure iron was noted, but to a lesser degree.

In agreement with the present authors, it was observed that grain size after recrystallization was greatest in the wires with least amount of cold working. For example, after annealing at 1475 degrees Fahr. (800 degrees Cent.),

grain up to $1/5$ of the cross section of the wire appeared in wires of all the alloys which had been cold drawn to only 2 per cent reduction in area. As the degree of drawing increased, the resulting grain size became finer.

The authors of the present paper have made such an excellent and thorough study of the effects of several alloying elements on recrystallization and hardness change that there seems to be no need for further research in the field they have covered.

Authors' Reply

The authors wish to thank Dr. Morral and Dr. Butler for their discussions. Replying first to the former, it is agreed that more data in the neighborhood of the intermediate reductions would help to fix more certainly the contour of the curve in Fig. 1. However, the evidence appears definite that the curve tends to become parallel to the horizontal between 40 and 75 per cent reduction before exhibiting a sharp inflection upward to 90 per cent. The important feature to which we wished to call attention was that the latter inflection disappeared when reduction was plotted on a logarithmic scale. Again in Fig. 3 it would probably have been better to average this curve for 20 per cent reduction between the points plotted for 900 and 1000 degrees Fahr. Some remarks with respect to the behavior of the various elements in affecting softening temperature may appear to be general, but the authors' intention was to portray a more or less general picture. It was felt at the present stage of the investigation that a specific, detailed treatment is not particularly desirable.

No specific reply is needed in respect to Dr. Butler's remarks. The agreement between his quoted results and the data presented in this paper is extremely gratifying to the authors.

FACTORS CONTROLLING GRAPHITIZATION OF CARBON STEELS AT SUBCRITICAL TEMPERATURES

BY CHARLES R. AUSTIN AND M. C. FETZER

Abstract

Previous researches in this series of papers had demonstrated the effects on graphitization of certain factors such as method of manufacture, chemical composition, martensitization prior to annealing, and internal oxidation of the steels. The present investigation throws more light on the effects of both mechanical treatment and of thermal treatment above the critical, prior to subcritical graphitization, and develops the role of environment on the progress of graphite formation.

The importance of martensitization in rendering the carbides unstable has been confirmed and the concept of increased solubility of a phase promoting graphitization appears to have received further direct support.

It has been shown that while compressive stresses have little effect, tensile stresses may profoundly increase carbide instability as revealed by subsequent tempering.

The role of subcritical graphite nucleation has also received attention. Thus it has been shown that while pretempering at 715 degrees Cent. (1320 degrees Fahr.) promotes carbide stability, an initial nucleating treatment at 670 degrees Cent. (1240 degrees Fahr.) may be followed by continued carbide dissociation on raising the temperature to 715 degrees Cent. (1320 degrees Fahr.).

The role of atmosphere has also received experimental consideration and it is clear that while the presence of oxygen in the environment used in tempering promotes graphitization, the increased susceptibility is dependent on prior austenitization followed by rapid cooling. Indeed the furnace atmosphere during austenitization has also been shown to play a part as demonstrated by peripheral rim effects on subsequent subcritical treatments.

The results appear to lend strong support to the suggested role of aluminum (calcium and silicon) oxide in promoting subcritical graphitization, when present in suitable form and suitable degree of dispersion.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. Of the authors, Charles R. Austin is professor of metallurgy, The Pennsylvania State College, State College, Pa., and M. C. Fetzer is research metallurgist, Carpenter Steel Company, Reading, Pa. Manuscript received June 15, 1944.

INTRODUCTION

PREVIOUS publications (1-6)¹ have shown that the factors influencing the graphitization of both pure hypereutectoid steels and commercial plain carbon steels are dependent on whether the reaction proceeds at temperatures above or below the critical (A_1). This investigation relates to a more comprehensive study of the factors controlling graphitization of eighteen 1 per cent plain carbon steels at subcritical temperatures. Previous discussions pertaining to the subcritical graphitization of the commercial steels permit the following statements:

1. Rate of dissociation of cementite is a maximum at about 670 degrees Cent. (1240 degrees Fahr.) in steels which are prone to graphitization.

2. The reaction rate is a maximum when the steel has been quenched previously in order to produce martensite.

3. Commercial carbon steels appear to exhibit marked differences in tendency to graphitization although the variation in chemical analysis may be quite small and well within the usual commercial limitations. The reason for the difference in behavior appears to be dependent on the mode of deoxidation. This factor relates particularly to deoxidation with metallic aluminum and to the stage in the steel making practice at which the aluminum is added. With the steel in a suitably partially deoxidized state, the addition of aluminum in the mold is most likely to promote what may be termed "temper graphite." The instability of the carbides has been ascribed to the presence of alumina in a certain physical state.

4. Some otherwise stable steels can be made to graphitize by internal oxidation. Thus tempering in an oxidizing bath presumably oxidizes certain metallic components of the steel, such as aluminum and silicon, to form nucleating oxides. This oxidizing atmosphere also accelerates the graphitization of otherwise unstable steels.

5. The particles of graphite are strung out in the direction of rolling and thus give rise to a banded structure. This banding is obliterated or masked when subcritical annealing is carried out under an oxidizing environment owing to the random location of nucleating particles produced by internal oxidation.

¹The figures appearing in parentheses refer to the bibliography appended to this paper.

PRESENT INVESTIGATION

The purpose of the present investigation was to throw more light on the effects of both mechanical treatment, and of thermal treatment above the critical, prior to subcritical graphitization, and to study further the role of environment (atmospheric conditions) on the progress of graphite formation.

It has already been demonstrated that prequenching to martensite may accelerate the reaction causing graphitization, but it is not yet clear why such pretreatment renders the cementite more prone to dissociation. Factors which may be cited relate to the fineness of the carbide dispersion which results on tempering martensite, to the presence of internal cracks or strain within the steel, or to some solution effect dependent on retention by quenching of a constituent in the austenite followed by precipitation, on subcritical annealing, in a form favorable for graphite nucleation. Furthermore, the role of this latter factor, since internal oxidation during prolonged tempering may promote graphitization, could be either major or minor, depending on whether the steels prior to dispatch for service conditions had been cast and fabricated under conditions favorable or unfavorable to the formation of this constituent.

MATERIALS

Eighteen plain carbon steels of about 1 per cent carbon and containing similar amounts of each of the other common elements usually found as impurities were obtained in the form of $\frac{3}{8}$ -inch diameter bar stock from various manufacturers. The method of manufacture and chemical analysis are given in Table I.

The steels have been designated by the letters H to Z and previous work has shown (5) that ten steels, listed H to R, inclusive, were resistant to carbide dissociation when tempered in covered lead (nonoxidizing conditions) at 670 degrees Cent. (1240 degrees Fahr.), whereas the group S to Z, inclusive, graphitized to varying degrees. With the exception of steels I and R, even the so-called stable steels exhibited some graphitization on prolonged tempering under oxidizing conditions in uncovered lead.

Table I
Manufacture and Chemical Analysis¹

Steel	Per Cent													Kind
	C	Si	Mn	S	P	Cr	Ni	Cu	Oxygen over that in Al ₂ O ₃ (calc.)	N	Al (metallic)	Al ₂ O ₃	Oxygen (total)	
H	1.08	0.33	0.36	0.017	0.020	0.11	0.04	0.12	0.0022	0.0065	0.010	0.006	0.005	Basic arc electric
I	1.02	0.27	0.22	0.010	0.014	0.09	0.04	0.13	0.0051	0.0070	0.005	0.004	0.007	Basic arc electric
J	1.03	0.27	0.31	0.020	0.017	0.06	0.05	0.10	0.0011	0.0045	None	0.002	0.002	Basic arc electric
K	1.00	0.45	0.30	0.022	0.020	0.06	0.06	0.13	0.0125	0.0070	None	0.001	0.013	H. F. induction ²
L	1.07	0.31	0.26	0.010	0.020	0.09	0.06	0.15	0.0061	0.0059	0.003	0.004	0.008	H. F. induction ²
M	1.00	0.20	0.18	0.021	0.017	0.08	0.04	0.17	0.0017	0.0093	0.010	0.007	0.005	Basic arc electric
N	1.12	0.20	0.25	0.013	0.017	0.04	0.13	0.13	0.0002	0.0101	0.007	0.008	0.004	Basic arc electric
O	1.06	0.13	0.19	0.010	0.016	0.03	0.06	0.13	0.0017	0.0097	0.010	0.008	0.0055	Basic arc electric
P	1.07	0.14	0.22	0.010	0.017	0.07	0.07	0.15	0.0018	0.0101	0.003	0.006	0.0045	Basic arc electric
R	1.01	0.28	0.34	0.021	0.010	0.06	0.10	0.08	0.0021	0.0034	0.001	0.004	0.004	Induction ³
S	1.03	0.21	0.24	0.017	0.017	0.04	0.04	0.15	0.0023	0.0104	0.004	0.006	0.005	Basic arc electric
T	1.01	0.21	0.21	0.019	0.012	0.10	0.08	0.13	0.0002	0.0042	0.003	0.007	0.0035	Basic open hearth
U	1.02	0.20	0.27	0.016	0.013	0.11	0.10	0.16	None	0.0042	0.020	0.009	0.0035	Basic open hearth
V	1.03	0.20	0.20	0.023	0.013	0.11	0.06	0.13	None	0.0034	0.002	0.008	0.0035	Basic arc electric
W	1.03	0.21	0.18	0.020	0.010	0.09	0.03	0.19	None	0.0039	0.018	0.010	0.0045	Basic open hearth
X	0.99	0.24	0.22	0.017	0.015	0.04	0.02	0.11	0.0003	0.0084	0.019	0.011	0.0055	Electric
Y	1.03	0.22	0.20	0.014	0.012	0.05	0.02	0.06	0.0002	0.0031	0.004	0.009	0.0045	Electric
Z	0.98	0.19	0.20	0.017	0.014	0.04	0.04	0.04	0.0023	0.0014	0.004	0.010	0.007	Electric

¹ Molybdenum and cobalt were reported as 0.0 per cent and vanadium as less than 0.01 per cent on all 18 steels. Traces of titanium were reported on steels R, V and Z but it was nil in the other 15 steels.

² Magnesia crucible.

³ Acid crucible, no slag.

EFFECT OF PRIOR HEAT TREATMENT
ABOVE THE CRITICAL ON REACTION TO GRAPHITIZATION

In an effort to determine more precisely the role of microstructural condition obtaining in the steels, prior to a graphitizing treatment at 670 degrees Cent. (1240 degrees Fahr.) in covered lead, samples of most of the commercial steels were first treated in the manner indicated in Table II. It will be noted that these tabulated

Table II
Indicating the Various Heat Treatments Given the Steels Prior to the
Subcritical Graphitizing Treatment at 670 Degrees Cent.

Nature of Treatment	Heat Treatment Prior to Tempering			Stress Relief Temp. °C.	Time of Tempering At 670 °C.
	Annealing Temp. °C.	Time Held At Temp.	Heating Medium		
As-received	1000	1 hour	Charcoal	None	600 hours
Furnace-cooled 2 °C. per minute	1000	1 hour	Charcoal	None	600 hours
Normalized	1000	1 hour	Charcoal	None	600 hours
Water-Quenched	1000	1 hour	Charcoal	200	
	850	10 min.	Air	200	1/2, 1, 5, 25,
	850	24 hr.	Charcoal	200	125, 272,
	800	24 hr.	Charcoal	200	and 600
	750	10 min.	Air	200	hours
Oil Quench	1000	1 hour	Charcoal	200	125 hours
	850	15 min.	Charcoal	200	125 hours
Water Quench	850	15 min.	Air	200	125 hours
				300	125 hours
Repeated Water Quench . .	850	15 min.	Charcoal	400	125 hours
				200	125 hours

data include as great a variety of prior treatments as practicable, ranging from the very slow furnace cooling from above the critical to water quenching after a variety of supercritical treatments. The "as-received" condition was, of course, dependent on the final operation given by the manufacturer. Hardness data, checked by microscopic examination, showed that all steels except M, S, T, U, V, and W were in the spheroidized condition. Those listed as exceptions were pearlitic.

In order to prevent scaling and serious decarburization, the prior heating was usually conducted under a protective layer of charcoal. However, the heating medium used is stated in the table along with the time of tempering at 670 degrees Cent. (1240 degrees Fahr.).

The effects of these various treatments on the response to softening on tempering at 670 degrees Cent. (1240 degrees Fahr.) have been shown in Table III. The difference in Rockwell "B" hardness values listed in the columns "before" and "after" tempering may be indicative of two reactions—spheroidization and softening dependent on graphitization. No hardness values were necessary as indicative

Table III
Showing Response to Softening, as Indicated by Rockwell "B" Hardness Values, When Tempered at 670 Degrees Cent. in Covered Lead.
Details of the various pretreatments are shown in Table II.

Steel	As-Received		Furnace-Cooled		Normalized		Tempered after Water Quench from		Tempered after—		Oil Quench from	
	Before	After*	Before	After*	Before	After*	850°— 10 min.*	850°— 24 hr.*	100°— 1 hr.*	1000°— 1 hr.†	1000°— 1 hr.†	850°— 15 min.†
H	83	82	97	91	110	90	87	86	88	93	93	88
I	90	86	95	88	108	87	85	83	82	85	85	88
J	84	82	97	90	109	89	85	86	87	91	91	88
K	97	85	99	90	110	89	86	85	84	87	87	88
L	84	83	98	89	109	89	85	84	85	90	90	89
M	98	89	96	89	109	88	87	87	85	91	91	89
N	71-82	81	96	88	108	88	84	84	84	87	87	87
O	81	77	95	87	108	86	85	84	80	85	85	85
P	81	81	95	88	108	87	84	85	85	86	86	86
R	87	83	96	88	109	88	86	86	83	86	86	86
S	100	86	96	88	109	87	52-57	49-63	54-61	63	65	87
T	98	48-62	94	85	108	86	54	55	55-63	68-73	63	87
U	98	87	96	87	108	87	63-67	63	66-78	79	73	90
V	99	53-66	95	86	107	86	48-59	49-59	56-65	71	65	66
W	98	46	96	61-83	108	86	45	47	52	60	48	58
X	85	81	94	86	106	85	45	47	47	65	55	66
Y	85	55	97	51	108	47-69	49	45	50	55	46	48
Z	82	45-64	93	35-47	105	80	39-43	39	40-46	36-43	40	42

*Tempered for 600 hours at 670 degrees Cent. in Covered Lead
†Tempered for 125 hours at 670 degrees Cent. in Covered Lead

of the hardness of the specimens after water or oil quenching since they were essentially martensitic.

With the prior structure in the "as-received" condition, steels T, V, W, Y, and Z exhibited marked tendency toward graphitization as indicated by change in hardness as a result of tempering 600 hours at 670 degrees Cent. (1240 degrees Fahr.). Steels T, V, and W were received in a fine pearlitic condition while steels Y and Z had been spheroidized by the manufacturer. Microscopic examination revealed slight graphitization in the pearlitic steels S and U and in the spheroidized steel X.

In order to promote a coarse pearlitic structure in all samples, the steels were placed in a pot, packed with charcoal, and cooled from 1000 degrees Cent. (1830 degrees Fahr.) at a rate of about 2 degrees Cent. per minute in the range 800 down to 650 degrees Cent. (1470 to 1200 degrees Fahr.). It was interesting to note that series S to Z, which previous tests had shown to be graphitizers, were slightly softer after furnace cooling than the stable series of steels (H to R). The hardness data obtained after 600 hours at 670 degrees Cent. (1240 degrees Fahr.) (Table III) showed clearly that only steels W, Y, and Z profoundly graphitized. Microscopic examination showed some carbide dissociation in steels S, T, Y, and X but steel U now appeared to be resistant to graphite formation at 670 degrees Cent. (1240 degrees Fahr.).

In general then, slow furnace cooling resulting in pearlite rendered the steels more stable on tempering at 670 degrees Cent. (1240 degrees Fahr.). However, steels Y and Z softened slightly more from a prior pearlitic structure resulting from furnace cooling, than from a commercial spheroidizing treatment. It is, of course, possible that residual cold work developed in the "hot-worked" steels may have had some effect on the greater tendency toward graphitization (steels T, V, and W) in some of the "as-received" pearlitic steels as compared with the pearlitic structure developed from slow furnace cooling.

To promote a fine pearlitic structure the samples were heated in a pot containing charcoal and air-cooled (normalized) after 1 hour at 1000 degrees Cent. (1830 degrees Fahr.). The hardness data obtained after 600 hours at 670 degrees Cent. (1240 degrees Fahr.) are perhaps surprising since only one steel (Y) showed marked graphitization, as indicated by a large decrease in hardness. Even with the microscopic examination only one other steel (Z) showed

any evidence of carbide dissociation. Evidently, normalizing had a more stabilizing effect on the carbides than slow furnace cooling of the steels.

The effect of prior quenching on acceleration of the process of graphitization has been established in earlier cited publications. From Table II it will have been noted that studies are here included to permit an analysis of any effect on graphitization dependent on vary-

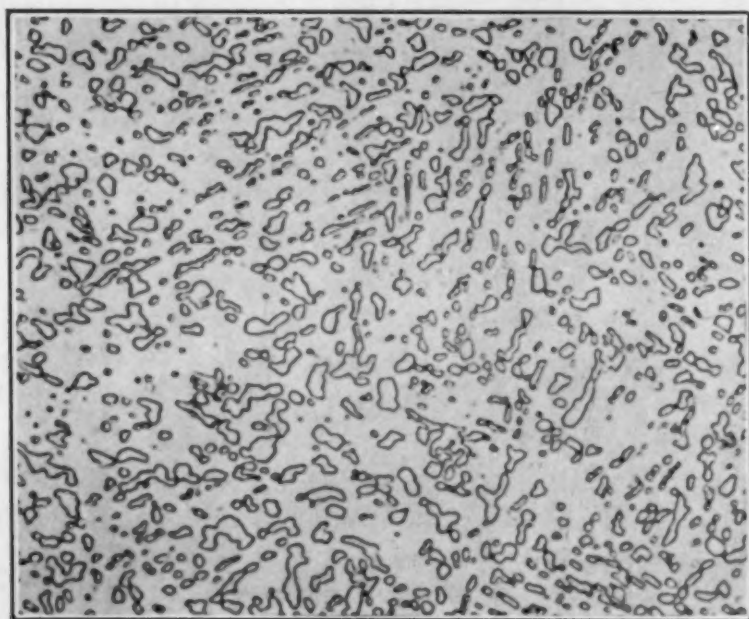


Fig. 1—Section of Steel H Showing Complete Resistance to Graphitization After Quenching from 1 Hour at 1000 Degrees Cent. (1830 Degrees Fahr.) Followed by Tempering at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in Covered Lead. Etched in Picral. $\times 1000$.

ing the austenitizing temperature and on time at temperature, prior to quenching for development of the martensitic structure. Immediately after quenching, the samples were stress relieved at 200 degrees Cent. (390 degrees Fahr.) and then tempered in covered lead at 670 degrees Cent. (1240 degrees Fahr.) for intervals of $\frac{1}{2}$, 1, 5, 25, 125, 272, or 600 hours. Hardness determinations were made on the transverse sections of the bar and the data obtained after 600 hours' treatment are recorded in Table III.

All quenched samples were examined under the microscope. After 1 hour at 1000 degrees Cent. (1830 degrees Fahr.) complete solution of carbides had been effected with a resulting coarse austenitic grain size. After 10 minutes at 850 degrees Cent. (1560 degrees Fahr.), most of the steels showed some evidence of incom-

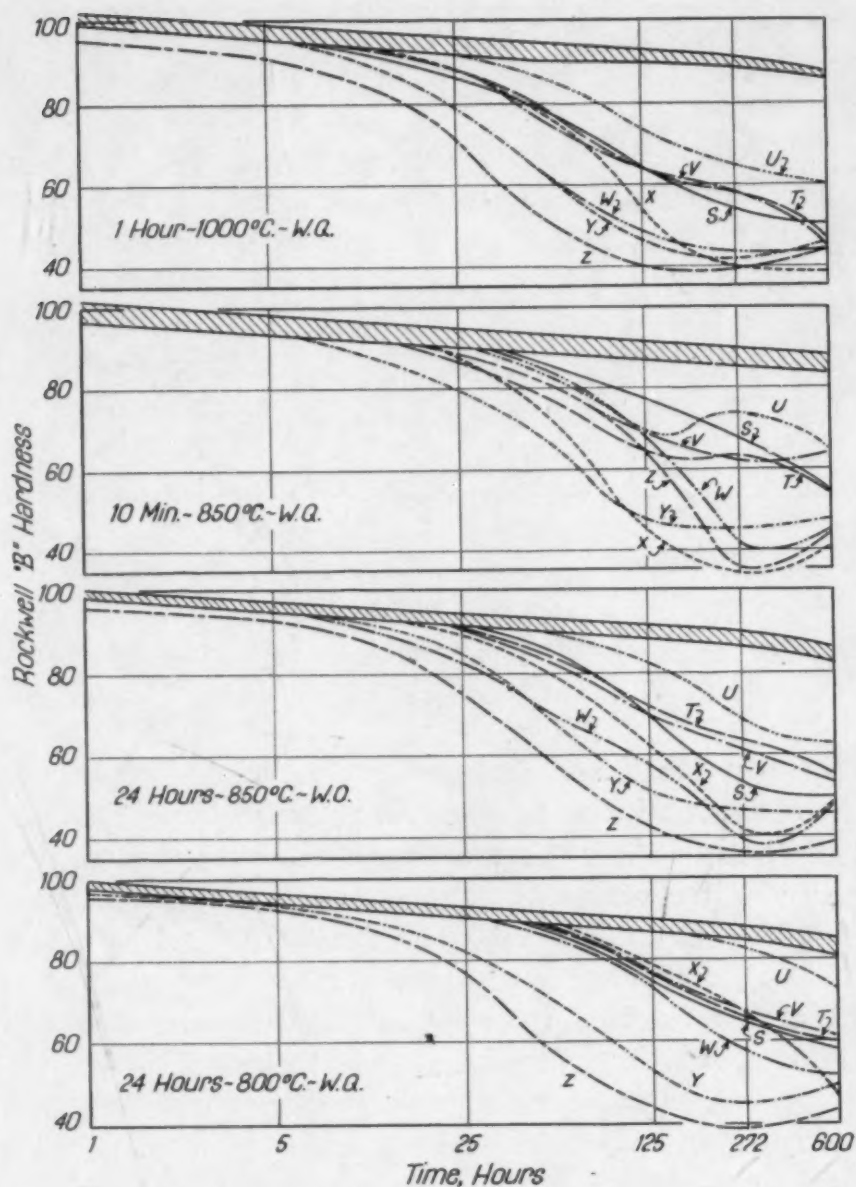


Fig. 2—Showing the Effect of Tempering for 600 Hours at 670 Degrees Cent. (1240 Degrees Fahr.) in Covered Lead After Four Different Austenitizing Treatments Followed by Water Quenching. The shaded portions on the hardness-time graphs indicated the limited spread in the ten stable steels which resist graphitization. The behavior of each of the eight unstable steels is shown by an individual plot.

plete carbide solution, and those with the slightly higher carbon content had a few cementite spheroids, even after 24 hours' holding at temperature. With a 24-hour treatment at 800 degrees Cent. (1470 degrees Fahr.), excess cementite in a martensitic matrix was observed in all the steels. The 10-minute treatment at 750 degrees Cent. (1380 degrees Fahr.) was selected as the austenitizing treat-

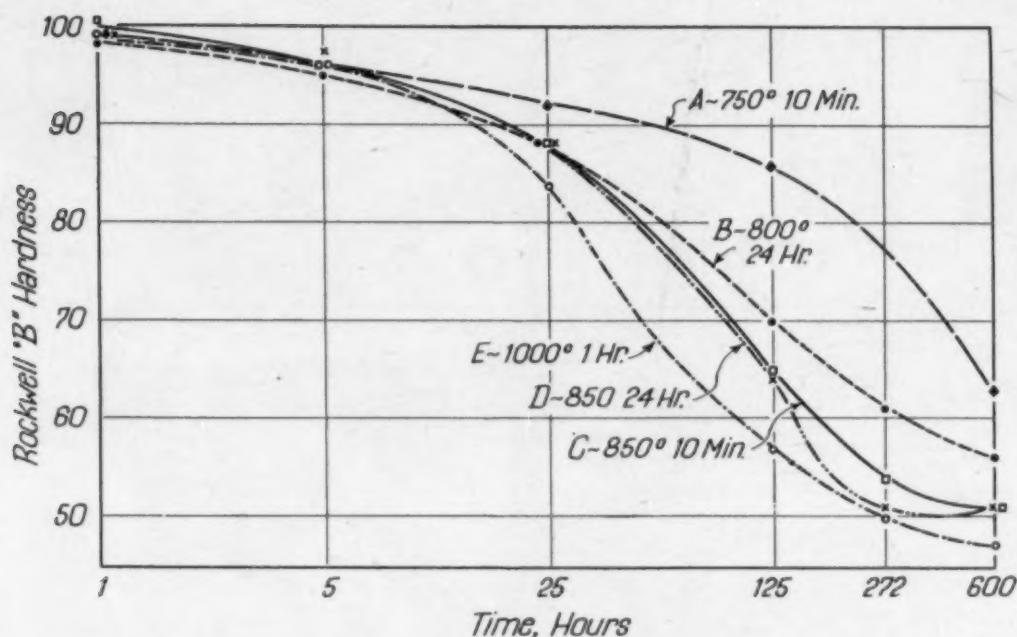


Fig. 3—Semi-Logarithmic Plot of the Average Hardness Values, Obtained from the Five Water Quenching Treatments Listed in Table II, at Successive Time Intervals, for Each of the Eight Unstable Steels.

ment likely to produce a martensite with minimum internal stress.

Steels H to R inclusive all appeared to resist graphitization irrespective of which of the five austenitizing treatments had been used. The stability of the cementite was confirmed in each instance by metallographic examination. A typical spheroidized structure is illustrated in Fig. 1, which shows the graphite-free microstructure of steel H after quenching from 1000 degrees Cent. (1830 degrees Fahr.) and tempering for 600 hours at 670 degrees Cent. (1240 degrees Fahr.). A semi-logarithmic plot showing the gradual but limited softening of these steels as a function of time is given in Fig. 2 for four of the five austenitizing treatments. There appears to be a slight decrease in ultimate hardness after 600 hours as the quenching temperature is lowered from 1000 to 800 degrees Cent. (1830 to 1470 degrees Fahr.). However, the linear relation between hardness and log tempering time of these 10 stable steels confirms the behavior at subcritical temperature established by one of the present authors some years ago.

The behavior of the eight unstable steels (S to Z, inclusive) as represented by the end hardness values (after 600 hours) is reported in Table III, and the relation of hardness to time at tempering temperature is shown in Fig. 2. The general order of susceptibility of graphitization of these steels does not appear to be markedly affected

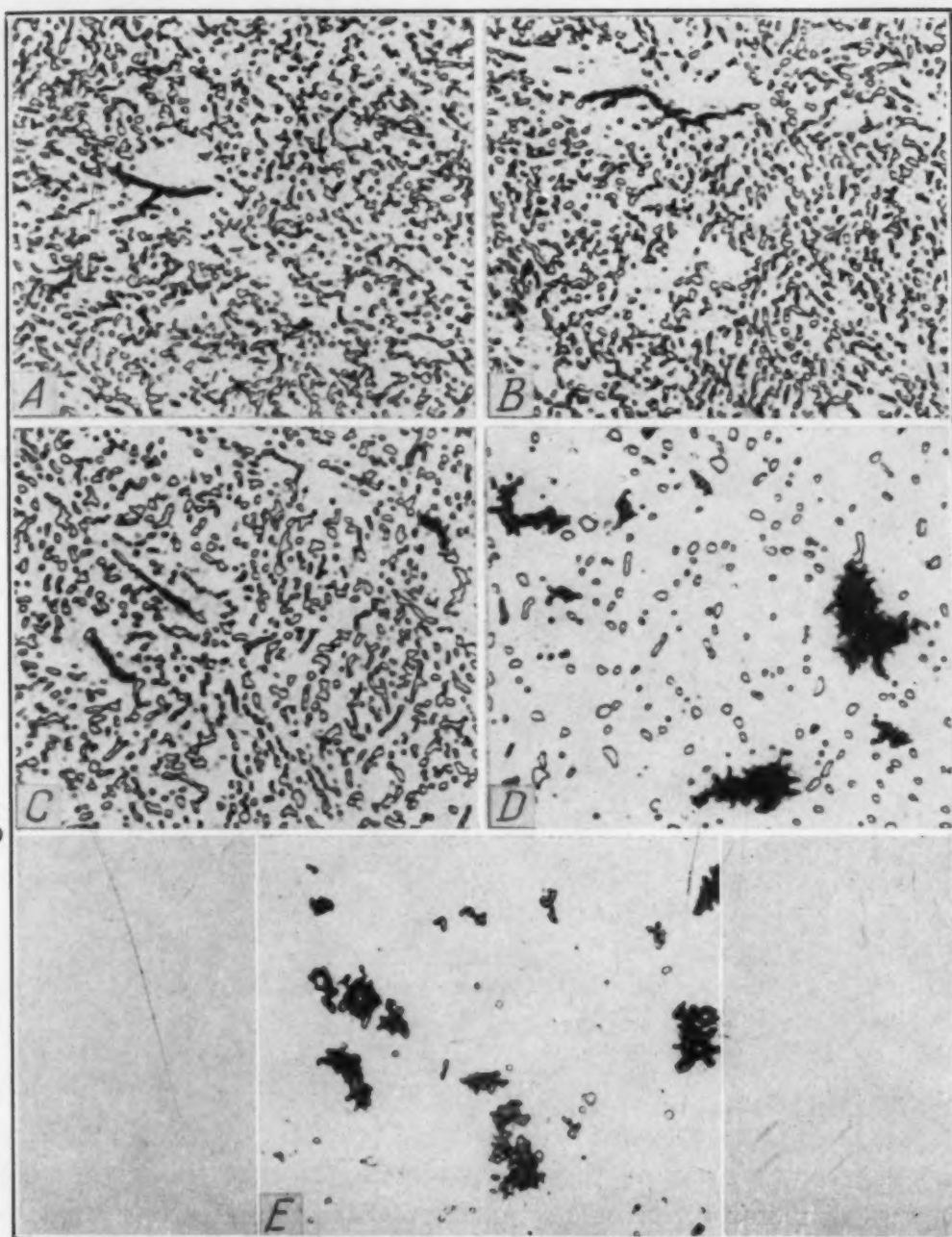


Fig. 4—Sections from Steel Z Showing Progress of Graphitization as a Function of Time up to 125 Hours on Tempering in Covered Lead at 670 Degrees Cent. (1240 Degrees Fahr.) After Quenching in Water from 1 Hour Austenitizing at 1000 Degrees Cent. (1830 Degrees Fahr.). Etched in Picral. $\times 1000$.
Time of Tempering.....A. $\frac{1}{2}$ hour. B. 1 hour. C. 5 hours.
D. 25 hours. E. 125 hours.

by prior austenitizing conditions. Steel U maintains the slowest rate of graphitization while steel Z is usually the one most prone to initiation of graphitization after only a few hours at 670 degrees

Cent. (1240 degrees Fahr.) and to complete graphitization after about 250 hours at temperature.

In order to check any statistical tendency of variation in prior austenitizing treatment on cementite dissociation, the average hardness values at successive time intervals of the eight graphitizing steels for each of the five quenching treatments have been plotted in Fig. 3.

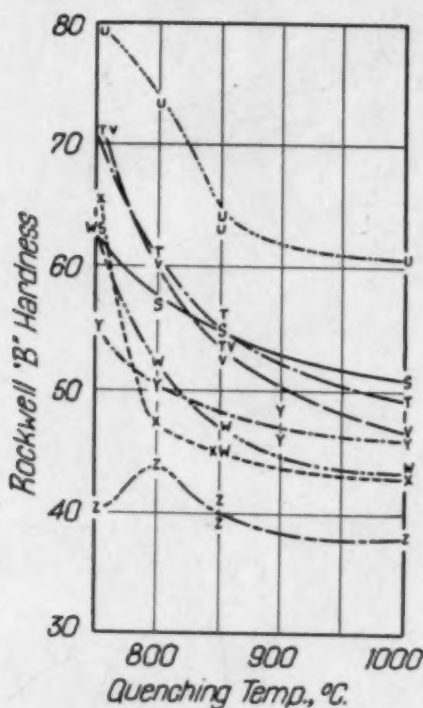


Fig. 5—Illustrating Progressive Effect of Increased Austenitizing Temperature Prior to Water Quenching on the Degree of Graphitization, as Indicated by Change in Hardness, After Tempering 600 Hours at 670 Degrees Cent. (1240 Degrees Fahr.).

The general reaction of these steels indicates that the higher the soaking temperature prior to quenching, the more prone are the steels to carbide dissociation. Time at austenitizing temperature appears to be relatively unimportant.

The progress of graphitization as revealed by the etched microstructure as a function of time after quenching from 1000 degrees Cent. (1830 degrees Fahr.) and annealing in covered lead at 670 degrees Cent. (1240 degrees Fahr.) is typified by the behavior of steel Z illustrated at 1000 diameters in Fig. 4.

Using the final hardness values for the various steels after tempering for 600 hours at 670 degrees Cent. (1240 degrees Fahr.)

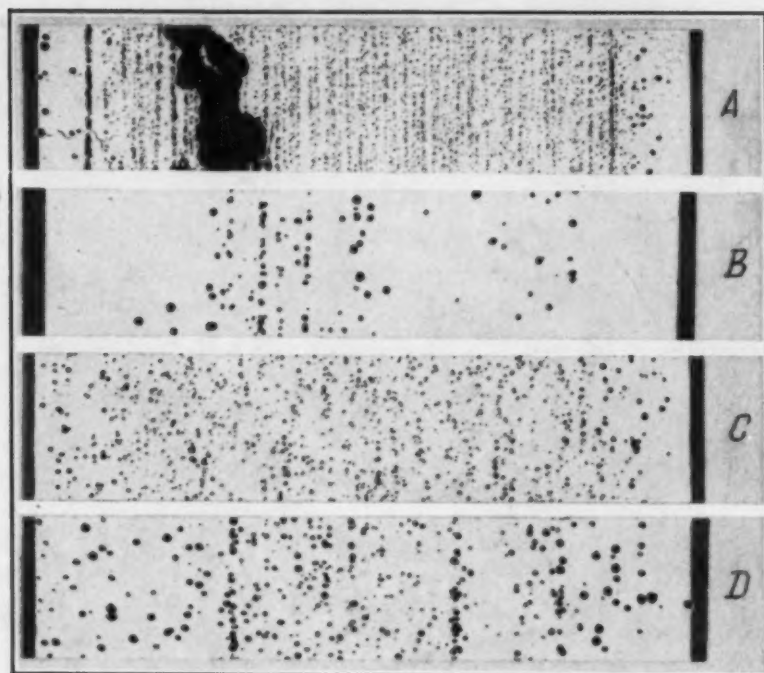


Fig. 6—Macrosections Taken from Steel Y Showing the Effect, on Distribution and Size of Graphite Particles, of Different Rates of Cooling Following Austenitizing for 1 Hour at 1000 Degrees Cent. (1830 Degrees Fahr.) Prior to Tempering for 600 Hours at 670 Degrees Cent. (1240 Degrees Fahr.) in Covered Lead. Unetched. $\times 7$.

A. Water-quenched.
C. Furnace-cooled.

B. Air-cooled (normalized).
D. As-received.

as an index of resistance to carbide dissociation, the importance of austenitizing temperature is well brought out in Fig. 5. Naturally, this progressive effect of increase in austenitizing temperature on subsequent subcritical graphitization is best revealed in the more stable of the so-called unstable steels, such as U, T, and V. The carbides in steel Z are so unstable that complete softening is effected in 600 hours at 670 degrees Cent. (1240 degrees Fahr.), even after austenitizing 10 minutes at 750 degrees Cent. (1380 degrees Fahr.).

In reviewing the reason for increased rate and degree of graphitization as a function of increase in austenitizing temperature from just above the A_{c1} to a temperature exceeding the A_{cm} , the following factors may need consideration: The higher the quenching temperature:

- (a) The higher the carbon content of the martensite.
- (b) The less the amount of free cementite.
- (c) The probable decrease in any residual graphite nuclei which may have been present from previous treatments.

(d) The greater the homogenization of the austenite with tendency for increase in austenitic grain size.

(e) The greater the internal stress with concomitant tendency for the formation of microcracks, as a result of quenching.

(f) The probably increased solution concentration of undefined elements or compounds which may promote carbide

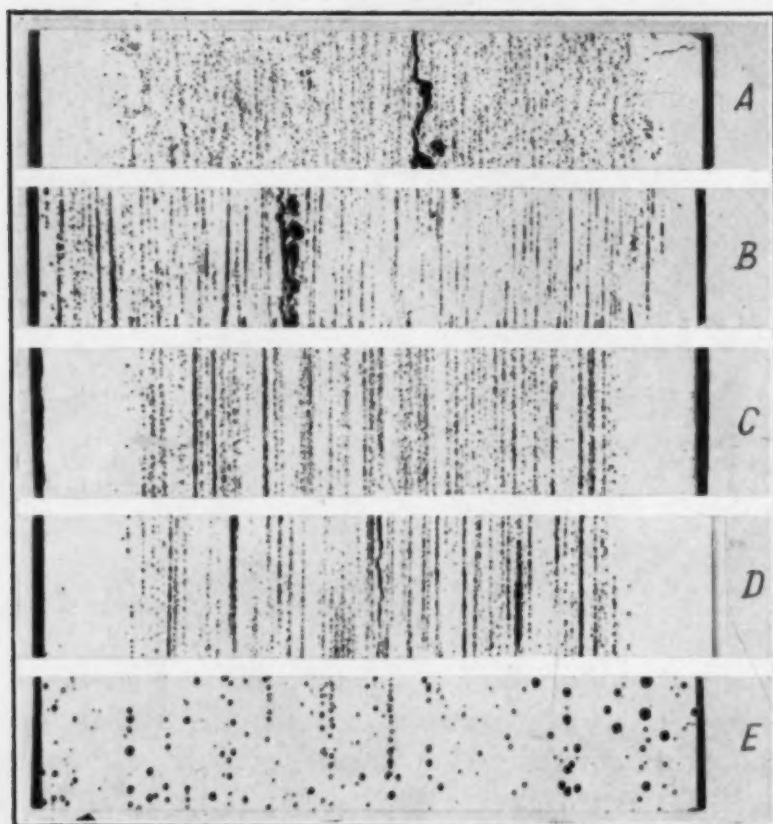


Fig. 7—Macrosections Taken from Steel Y Showing Effect, on Distribution and Size of Graphite Particles, of Different Austenitizing Treatments Followed by Water Quenching, Prior to Tempering for 125 Hours at 670 Degrees Cent. (1240 Degrees Fahr.) in Covered Lead. Unetched. $\times 7$.
A. 1 hour at 1000 Degrees Cent. B. 10 min. at 850 Degrees Cent.
C. 24 hours at 850 Degrees Cent. D. 24 hours at 800 Degrees Cent.
E. 10 min. at 750 Degrees Cent.

instability on subsequent precipitation in association with the cementite.

It is clear from the results depicted in Figs. 3 and 5 that a fine carbide dispersion, present in all quenched samples, is not the only factor increasing the rate of graphitization. Indeed, in certain instances, it might be deduced that degree of dispersion of the carbide

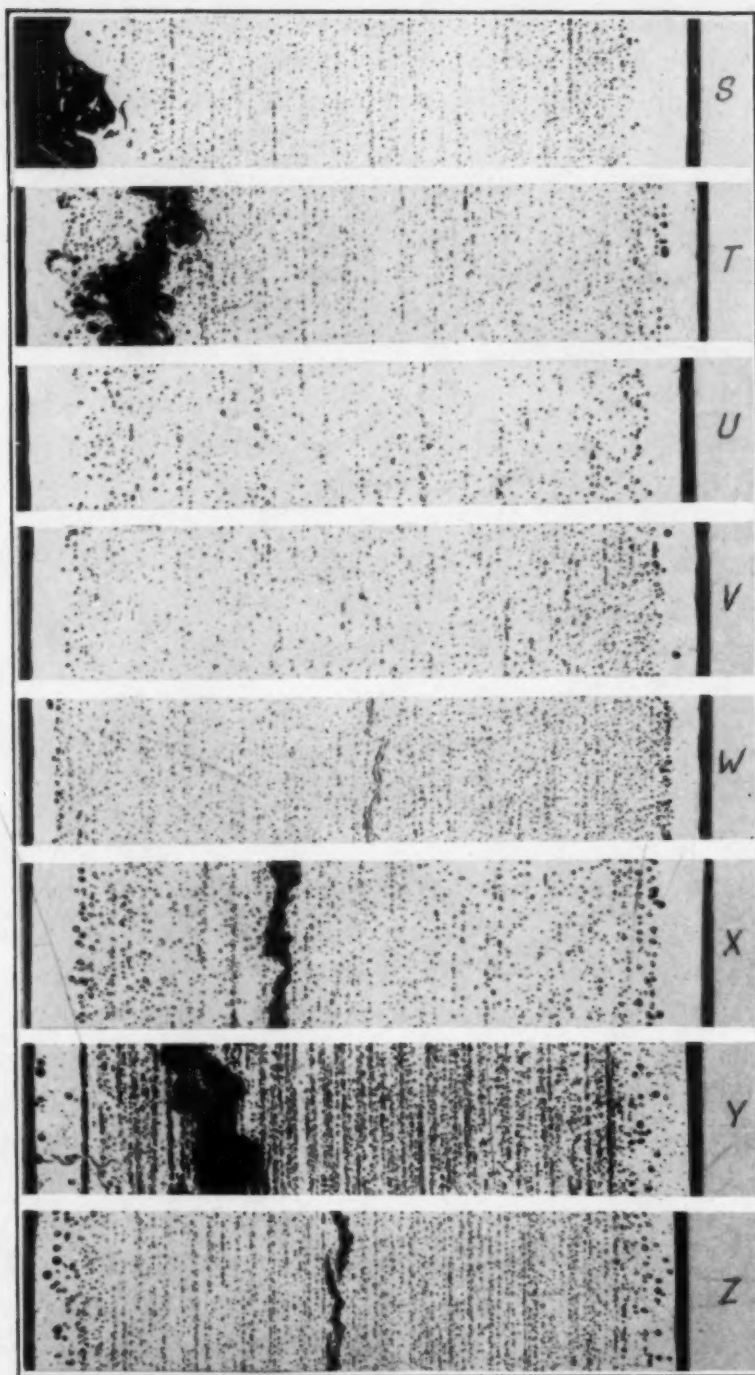


Fig. 8—Macrosections Taken from Each of the Eight Unstable Steels Showing the General Similarity, but Difference in Degree of Graphitization, After Water Quenching from 1 Hour at 1000 Degrees Cent. (1830 Degrees Fahr.) Followed by Tempering for 600 Hours in Covered Lead. Unetched. $\times 7$.

is unimportant. Thus steels T and W appear to be as completely graphitized after 600 hours at 670 degrees Cent. (1240 degrees

Fahr.), whether tempered in the "as-received" spheroidized condition or after martensitization by water quenching (Table II). However, the rate of graphitization may have been different for the two different prestructural states. Of the factors listed above, it would appear that increased strain associated with incipient microcracking and increased solubility of some substance promoting graphitization, both

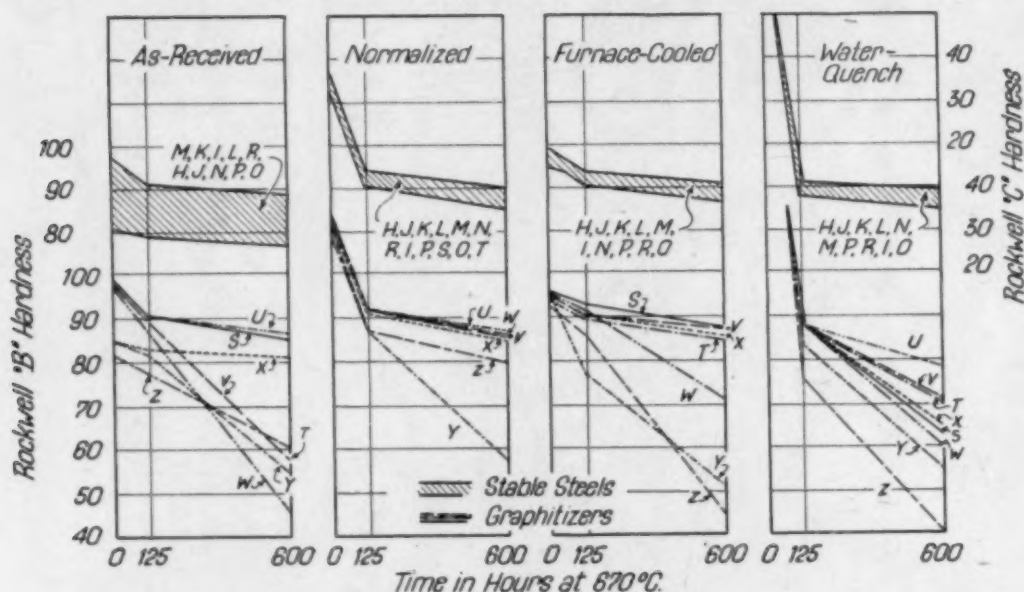


Fig. 9—Illustrating the Reaction of the Stable and Unstable Groups of Steels to Tempering up to 600 Hours at 670 Degrees Cent. (1240 Degrees Fahr.) in Covered Lead in the "As-Received," Normalized, Furnace-Cooled, and Water-Quenched Condition.

dependent on higher quenching temperature, may account for the observed change in carbide instability.

Attention should also be directed to graphite size and distribution dependent on the prior austenitizing treatment. The effect of cooling steel Y, a fairly strong graphitizer under the various conditions previously outlined, from 1000 degrees Cent. (1830 degrees Fahr.) at varying rates from water quench to furnace cool, is compared with the reaction of the "as-received" spheroidized condition to tempering for 600 hours at 670 degrees Cent. (1240 degrees Fahr.) in Fig. 6. These macrostructures at 7 diameters indicate maximum graphite nucleation in the water-quenched sample but there is no regular decrease in nucleation as the rate of cooling is progressively decreased. The reason for the irregularity in the hardness data taken on the sample which was air-cooled prior to tempering is clearly indicated. The directional distribution of the graphite, presumably associated with segregation, may also be noted.

The large dark area shown in Fig. 6A represents the appearance of a quenching crack.

In Fig. 7 the macrostructures indicate the nucleation characteristic for steel Y as a function of quenching temperature. Prolific nucleation is noted as a result of quenching from temperatures varying from 1000 to 800 degrees Cent. (1830 to 1470 degrees Fahr.). On quenching from 750 degrees Cent. (1380 degrees Fahr.) the graphite nodules resulting from tempering in covered lead for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) are large and relatively few in number. The absence of graphitization on the peripheral parts of the sample will be discussed later under a section on "rim effect."

The general similarity of behavior of all the graphitizing steels is illustrated in Fig. 8, showing the appearance of unetched macrosections at 7 diameters of steels S, T, U, V, W, X, Y, and Z after tempering for 600 hours in covered lead at 670 degrees Cent. (1240 degrees Fahr.), following a water quench from 1000 degrees Cent. (1830 degrees Fahr.). Quenching cracks are again evident in some of the samples.

In an attempt to group the results obtained in this section of the paper on tempering both the stable and unstable steels at 670 degrees Cent. (1240 degrees Fahr.) in covered lead after various prior microstructural conditions, hardness-tempering time graphs have been reproduced in Fig. 9. Since data for 125 and 600 hours' tempering only were available for the "as-received," normalized and furnace-cooled samples, these points alone were used in constructing all the graphs in this figure.

The consistent resistance to graphitization of the so-called stable steels, no matter what the prior heat treatment, is at once evident. Among the graphitizers, the more unstable steels appear to graphitize readily after each of the four different pretreatments, but the apparent greater resistance to carbide dissociation of steel Z after normalizing is noteworthy.

SUPPLEMENTARY PRETREATMENT STUDIES

From the data already presented, it appears that prior quenching of a steel may play an important role in graphitization. Whether this tendency is dependent on induced stresses or on a solution effect with possible subsequent precipitation is not clear. In order to study

further the role of internal stress, the following tests were conducted:

1. Oil quenching from 1000 degrees Cent. (1830 degrees Fahr.), followed by immediate tempering for 1 hour at 200 degrees Cent. (390 degrees Fahr.). This treatment might be assumed to promote less microcracking dependent on reduced internal stress.

2. Repeated water quenching from 1000 degrees Cent. (1830 degrees Fahr.), in order to observe if the probable increase in microcracking and internal strain would promote increased tendency toward graphitization.

3. Straining of the steel by cold working, either under tensile stress or by cold rolling.

4. Water quenching after 10 minutes at 850 degrees Cent. (1560 degrees Fahr.), followed by a pretempering at 715 degrees Cent. (1320 degrees Fahr.) for 50 hours in covered lead. Previous studies had established the relative resistance to graphitization at temperatures just below the critical of steels prone to rapid graphite formation at lower temperatures.

5. Water quenching the "as-received" steels after annealing for 1 hour below the critical (715 degrees Cent.), in order to observe any ferritic solution effect.

Brief comments will now be made on the effects of the above treatments on reaction to tempering for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) in covered lead:

1. All the unstable and three of the stable steels were oil-quenched from 1-hour anneal in charcoal at 1000 degrees Cent. (1830 degrees Fahr.), and then tempered 125 hours at 670 degrees Cent. (1240 degrees Fahr.). The hardness data (Table III) indicate the general similarity between oil and water quenching. Four of the steels (see Table III) were also oil-quenched after austenitizing only 15 minutes at 850 degrees Cent. (1560 degrees Fahr.). On tempering at 670 degrees Cent. (1240 degrees Fahr.) even the unstable steels resisted graphitization. Metallographic examination showed the 850-degree Cent. (1560-degree Fahr.) oil-quenched steels to be pearlitic. Thus, graphitization of at least two of the unstable steels (S and U) is dependent on prior martensitization of the structure or on the large stresses induced thereby and cannot be affected by the presence of simple quenching stresses alone.

These results seem to indicate that both an increased solid solution effect dependent on high temperature of pretreatment as well as on the presence of martensite, concomitant with internal strain, are necessary for promoting carbide instability on subsequent subcritical annealing.

2. For this study of the effect of repeated quenching and of low temperature pretempering on graphitization at 670 de-

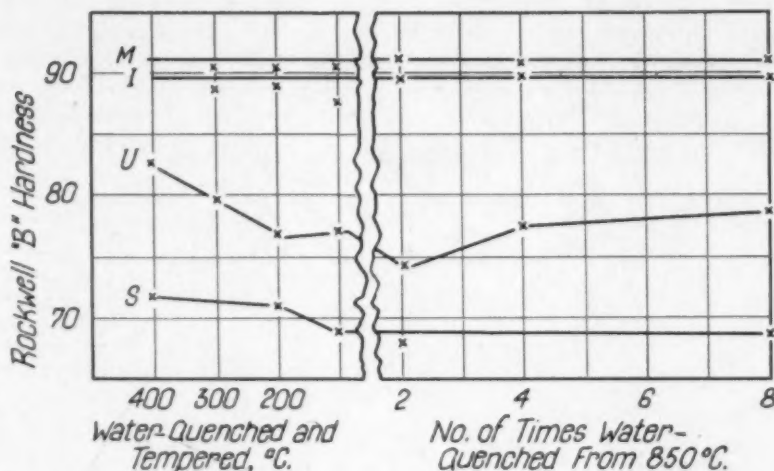


Fig. 10—Showing That Repeated Quenching from an Austenitizing Temperature of 850 Degrees Cent. (1560 Degrees Fahr.) Has Little Effect on Graphitization at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours. Pretempering at 300 and 400 Degrees Cent. (570 and 750 Degrees Fahr.) appears to render the unstable steels slightly less susceptible to graphitization at 670 Degrees Cent. (1240 Degrees Fahr.).

degrees Cent. (1240 degrees Fahr.) for 125 hours, two graphitizers (S and U) and two stable steels (I and M) were selected. Separate samples were quenched 1, 2, 4, and 8 times after 15 minutes at 850 degrees Cent. (1560 degrees Fahr.). Other samples were quenched once and then pretempered at 200, 300, or 400 degrees Cent. (390, 570, or 750 degrees Fahr.). The hardness values obtained after the 670-degree Cent. (1240-degree Fahr.) treatment in covered lead are shown in Fig. 10. Clearly, if repeated quenching results in any increase in internal strain or in the development of greater microcracking, these factors are not important in promoting graphitization. The increase in pretempering temperature prior to the graphitization appears to reduce cementite instability, particularly in steel U.

3. The cold work studies of both pearlitic and spheroidized structures consisted of cold rolling up to 33 per cent reduction and loading to fracture tapered tensile specimens.

For the cold rolling tests two stable (K and M) and three unstable steels (S, V, and Z) were machined flat on opposite sides of the $\frac{3}{8}$ -inch round to 0.20 inch thick. Steels K and Z were in the "as-received" spheroidized state, whereas steels M, V, and S were pearlitic. Four successive passes through the flat rolls provided 3, 6,

Table IV
Hardness Values Obtained After Tempering in Covered Lead for 125 Hours at 670 Degrees Cent. Subsequent to (a) Cold Rolling 33 Per Cent Reduction, and (b) Cold Deforming to Fracture in Tensile to About 30 Per Cent Reduction

Steel	As-Received		Furnace-Cooled	
	No deformation	Cold-Rolled 33% Reduction	No deformation	Stressed in Tension to Fracture
	Rockwell "B" after tempering for 125 hours at 670 °C.			
H	83	..	94	..
I	88	..	92	..
J	83	..	92	..
K	86	86	94	87
L	83	..	94	..
M	92	83	92	84
N	82	..	92	..
O	79	..	91	..
P	81	..	92	..
R	85	..	93	..
S	92	87	93	80
T	89	..	90	57
U	91	..	91	85
V	90	83	91	74
W	86	..	92	74
X	84	..	90	71
Y	82	..	77	66
Z	78	60	86	51

14, and 33 per cent reduction in thickness. All samples were annealed for 125 hours in covered lead at 670 degrees Cent. (1240 degrees Fahr.). The process of softening by tempering may involve both spheroidization and graphitization. Correlation of hardness data with metallographic examination leads to the conclusion that the behavior of the stable steels was unaffected by the rolling stresses. The unstable steels showed some but only minor increase in graphitization dependent on cold work. This is clear from Table IV.

The studies on the effect of tensile strain prior to tempering were more informative. Two of the stable steels (K and M) and all eight of the graphitizing steels were heated above the critical and furnace-cooled. Tensile bars were then machined from the $\frac{3}{8}$ -inch round stock with a tapered gage section $1\frac{1}{2}$ inches long varying in diameter from 0.27 inch to 0.32 inch. All bars were subject to tensile strain until fracture occurred at some recorded total stress. From this value it was possible to calculate the stress in pounds per square inch at different locations of the tapered gage section. After

tempering for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) and suitably grinding flats on the test samples, a series of hardness values was obtained on the Vickers hardness tester. The relation between final Vickers pyramid hardness after tempering and the tensile stress before tempering for the ten steels can be conveniently grouped into three typical curves (Fig. 11). The behavior of the

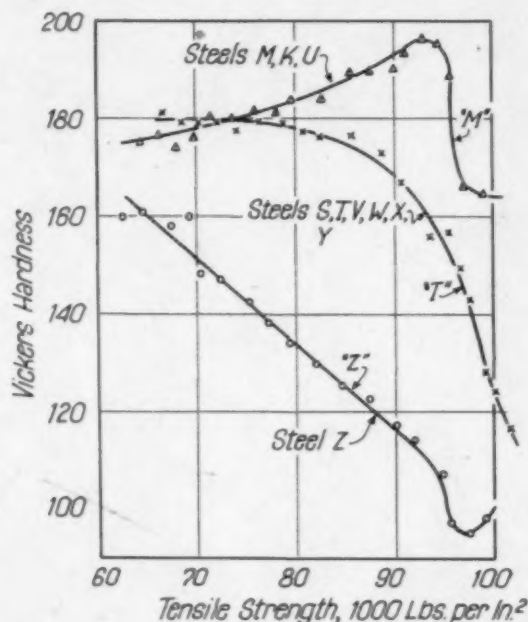


Fig. 11—Effect of Tensile Strain on the Pearlitic Structures of the Steels Prior to Graphitization Treatment at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in Covered Lead. Curve I (Steel T) is typical of the behavior of steels S, T, V, W, X, and Y. Curve II (Steel M) is typical of steels M, K, and U, and curve III indicates the markedly different response of steel Z.

two stable steels (K and M) and one graphitizer (U) showed a gradual increase in hardness (to 195 VPN) up to a region in the tapered tensile bar approximating 93,000 pounds per square inch pretempering stress. Thereafter the hardness dropped markedly to 165 VPN near the tensile fracture which resulted from 99,000 pounds per square inch. The curve was drawn from data obtained with steel M but steels K and U react quite similarly.

Metallographic examination showed complete absence of any graphitization at any section. Three photomicrographs (Fig. 12) taken from steel M after the 125-hour tempering at 670 degrees Cent. (1240 degrees Fahr.) show progressive degrees of spheroidization as the pretemper tensile stress was increased from 65,000 to

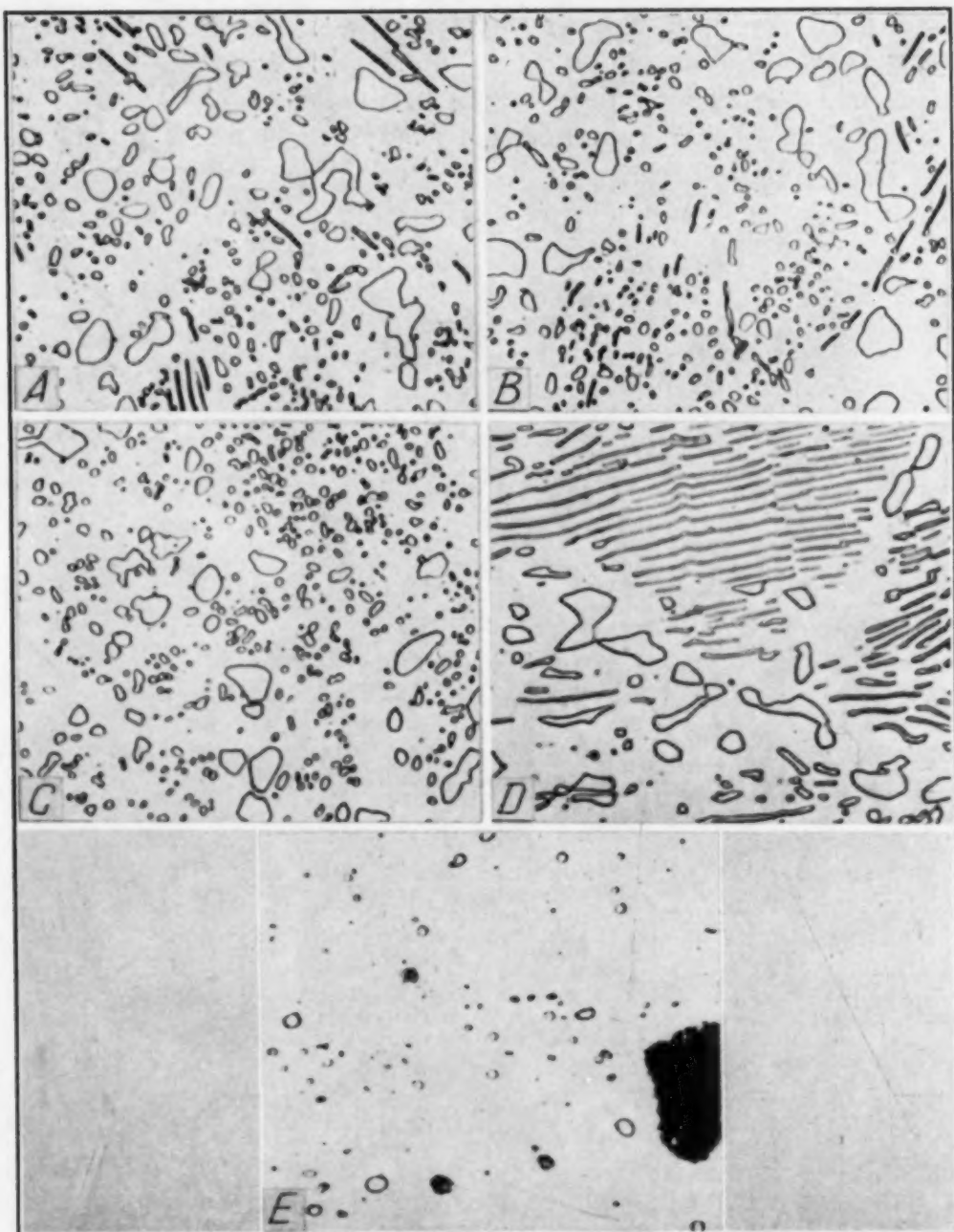


Fig. 12—Effect of Pretensile Strain of the Pearlitic Structures, on Subsequent Graphitization Treatment at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in Covered Lead.

12A, 12B, 12C. Steel M prestressed 65,000, 93,000 and 99,000 psi., respectively.

12D, 12E. Steel Z prestressed 73,000 and 100,000 psi., respectively. Etched. $\times 1000$.

93,000 and finally to 99,000 pounds per square inch. The increase in hardness up to 93,000 pounds per square inch can be assumed to be dependent on residual strain in the steel. The sudden drop near the fracture can be associated with the absence of any pearlite, and

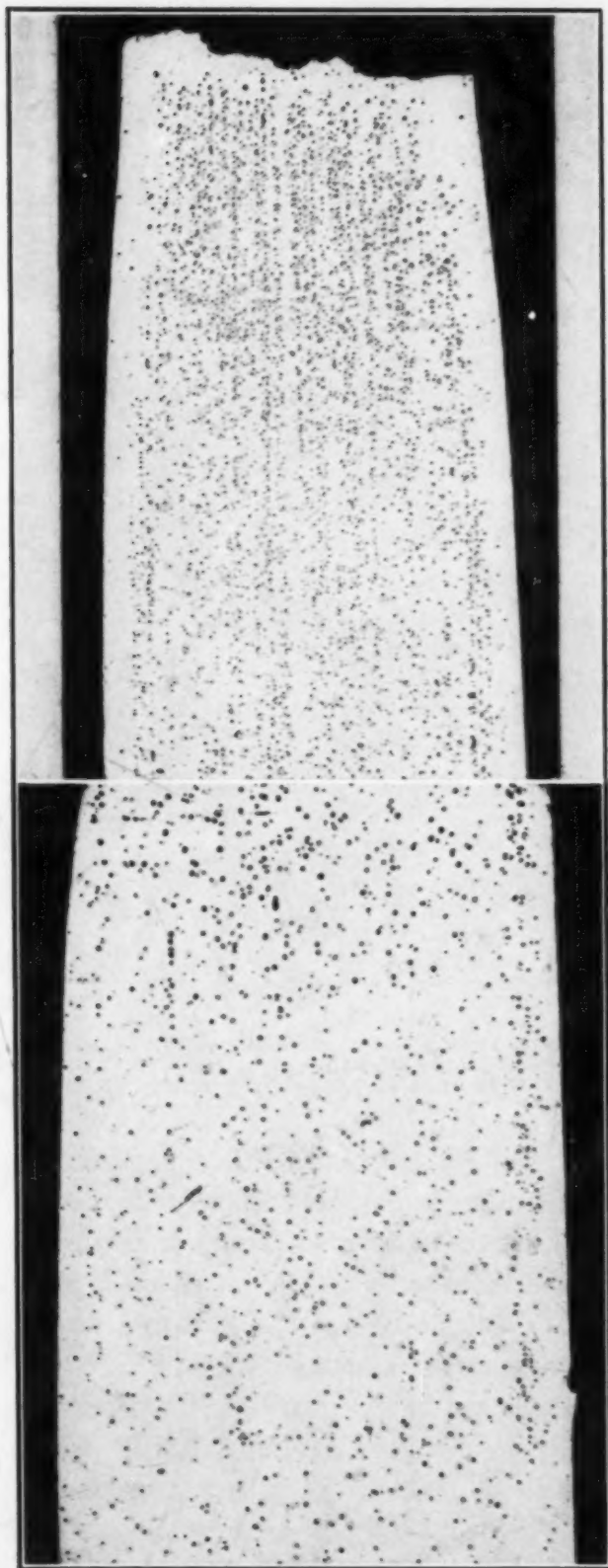


Fig. 13—Effect of Tensile Gradient on Steel Z on Subsequent Graphitization in Covered Lead at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours. Unetched. $\times 7$. (Upper)—Fractured End. (Lower)—Slightly Stressed End.

hence with the complete spheroidization of the pearlite resulting from the initial furnace cool from above the critical.

The general behavior of six of the eight unstable steels is represented by the plot for steel T. (Fig. 11). The lower stresses do

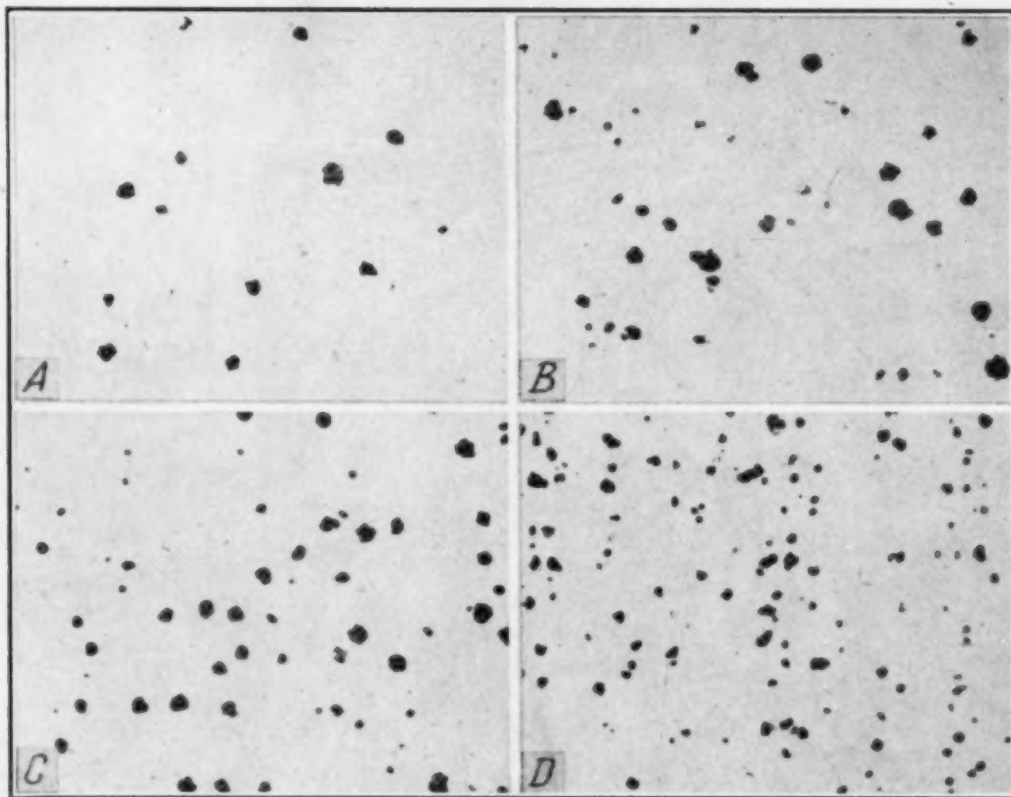


Fig. 14—Effect of Prior Tensile Stress on Steel Z on Subsequent Graphitizing Treatment at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in Covered Lead. 14A, 14B, 14C, and 14D. Unetched microsections taken from regions which were pre-stressed 70,000, 80,000, 90,000 and 97,000 psi., respectively. $\times 100$.

not have any marked effect on subsequent softening but as the pre-straining treatment exceeded a stress of 85,000 to 90,000 pounds per square inch the effect of temper softening is profound.

One of the steels, strongly graphitizing Z, reacted differently from any of the other alloys and the Vickers hardness-tensile relation is shown by a separate curve in Fig. 11. From metallographic examination, it seems that the linear relation is associated with progressive increase in quantity of graphite as the prestress was raised from about 65,000 to 100,000 pounds per square inch along the tapered sample. Two micrographs at 1000 diameters (Fig. 12) taken from locations stressed about 73,000 and 100,000 pounds per square inch (at fracture), respectively, clearly demonstrate the effect

of severe cold deformation on cementite dissociation in a pearlitic steel on prolonged holding at subcritical temperatures. Some idea of the effect of stress on graphitization of steel Z is portrayed in the macrosections (Fig. 13) taken from the lightly stressed (lower) and from the fracture end (upper) of the tapered section.

Microsections of the polished unetched tensile bar taken at 100 diameters (Fig. 14) show clearly the increase in graphite nucleation and amount of graphitization with increase in tensile strain, on tempering for 125 hours at 670 degrees Cent. (1240 degrees Fahr.). The hardness data close to the fracture obtained on the various steels after tempering have been included in Table IV.

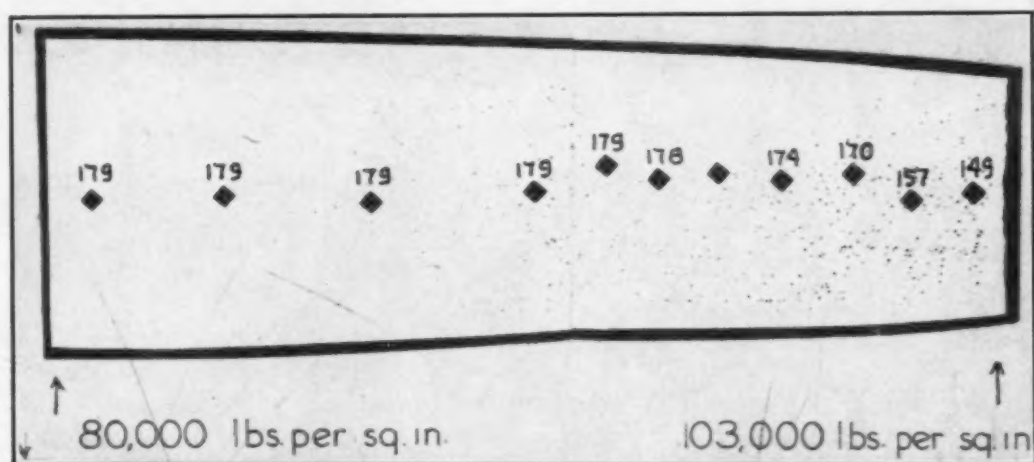


Fig. 15—Macrosection from the Complete Tapered Gage Section of Steel S Prestressed Over a Range 80,000 to 103,000 Psi. and Graphitized at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in Covered Lead. Initial structure pearlite. The VPN impressions and the graphite distribution may be noted. $\times 7$.

It has been pointed out in previous researches that hardness data must be supplemented with metallography in order to avoid erroneous conclusions. Thus, with most of the unstable steels, microexamination was required to establish a satisfactory qualitative evaluation of the presence of graphite and of the effect of stress or strain upon its formation. This was true for example with steel S.

Fig. 15 shows a macropicture at 7 diameters of a section of the complete gage length of specimen S. The progressive increase in graphitization with increasing stress is shown by the increase in number of graphite particles. However, it may be noted that there is no marked change in the size of the Vickers pyramid impression, and hence on Vickers hardness number, until a region close to the fracture is approached.

The results demonstrate that cold working by prestraining in tension may have a marked influence on accelerating the rate of graphitization. Cold work by flat rolling is relatively ineffective.

4. For the studies on the effect of pretempering at 715 degrees Cent. (1320 degrees Fahr.) for 50 hours, after water quenching from 10 minutes at 850 degrees Cent. (1560 degrees

Table V

Effect of Pretempering 50 Hours at 715 Degrees Cent. on Subsequent Graphitizing Anneal at 670 Degrees Cent., with Respect to One Stable and Seven Unstable Steels. The austenitizing treatment consisted of heating for 10 minutes at 850 degrees Cent. followed by water quenching. Both the 715 and 670-degree Cent. tempering treatments were conducted in a lead bath protected from oxidation

Steel	Rockwell "B" Hardness		
	Tempered at 715 °C. for 50 hr.		No pretemper
	No further treatment	125 hours at 670 °C.	125 hours at 670 °C.
I	89	88	88
S	91	90	77*
T	89	81*	70*
U	89	89	82*
V	88*	86*	68*
W	89*	82*	69*
X	87*	86*	65*
Z	83*	40*	38*

*Microscope indicates some graphite

Fahr.) prior to the regular treatment at 670 degrees Cent. (1240 degrees Fahr.) in covered lead for 125 hours, seven unstable steels (S, T, U, V, W, X, and Z) and one stable steel (I) were chosen. Previously, published work on these commercial steels had demonstrated resistance to graphitization at 715 degrees Cent.—just under the lower critical.

Metallographic examination of specimens subject to this 715-degree Cent. (1320-degree Fahr.) treatment confirmed that steels V, W, and Z were very slightly graphitized; steel Z, the most unstable, exhibiting possibly about 1 or 2 per cent cementite dissociation. The extent of graphitization in steels V and W was so slight that at 900 diameters only one field in ten showed any graphite particles. The stable steel, I, was free from graphitization and it appeared doubtful if any graphite had formed in steels S, T, U, and X.

After a subsequent temper for 125 hours at 670 degrees Cent. (1240 degrees Fahr.), examination under the microscope showed that steels S and U and the stable steel I were essentially graphite free, but T, V, W, X, and Z all graphitized slightly to varying degrees. Of these unstable steels, all but Z showed carbide dissociation had

proceeded no further than about 5 per cent to total graphitization, whereas steel Z was almost completely graphitized.

A general concept of the effect of the pretemper at 715 degrees Cent. (1320 degrees Fahr.), prior to tempering at 670 degrees Cent. (1240 degrees Fahr.), on this limited series of steels is shown by the change in hardness, from the values listed in Table V. The anneal at 715 degrees Cent. (1320 degrees Fahr.) causes little graphitiza-

Table VI
Indicating That Any Solution Effect Which May Result from Quenching of the Graphite Just Below the A_1 (715 Degrees Cent.) Compared with the Untreated "As-Received" Samples, Does Not Modify the Subsequent Graphitization Characteristics on Tempering for 125 Hours at 670 Degrees Cent. in Covered Lead

Steel	As-Received	Annealed at 715 °C. for 1 hour and Water-Quenched
	—Rockwell "B" after Tempering for 125 Hours at 670 °C.—	
H	83	83
I	88	89
J	83	93
K	86	90
L	83	82
M	92	92
N	82	80
O	79	80
P	81	81
R	85	84
S	92	92
T	89	89
U	91	91
V	90	91
W	86	91
X	84	83
Y	82	84
Z	78	80

tion in any of the steels, as previously reported, and has a decided effect in decreasing the rate of carbide dissociation on subsequent tempering at 670 degrees Cent. (1240 degrees Fahr.). Indeed, in the case of two of the so-called unstable steels, S and U, graphitization at 670 degrees Cent. (1240 degrees Fahr.) is practically inhibited completely by the 715-degree Cent. (1320-degree Fahr.) preanneal. This behavior is comparable to that obtained on tempering the "as-received" spheroidized steels.

Since it has already been demonstrated that the increase in particle size of the spheroidal cementite, obtained by the treatment at 715 degrees Cent. (1320 degrees Fahr.), is not responsible for any marked change in the rate of graphitization, it may be assumed then, that the pretreatment in some way modifies the nucleating effect of a foreign substance. This could relate to particle size or nature of any free alumina.

5. In the study on any ferritic solution effect, samples of all the steels were quenched after annealing the "as-received" alloys for 1 hour at 715 degrees Cent. (1320 degrees Fahr.). The samples were then tempered for 125 hours in covered lead at 670 degrees Cent. (1240 degrees Fahr.). The hardness data were compared with those obtained after similar temper-

Table VII

Showing Effect of Graphite Nucleation for 5 and 25 Hours at 670 Degrees Cent. on Subsequent Graphitization on Tempering for 100 Hours at 715 Degrees Cent., as Indicated by Rockwell "B" Hardness Values. For comparison purposes a subsequent tempering treatment at 670 degrees Cent. was also included. All samples were water-quenched after 1 hour at 1000 degrees Cent. and the tempering was conducted in a lead bath protected from oxidation

Steel	5-Hour Nucleation at 670 °C.		
	No Further Treatment	Followed by 100 hr. at 670 °C.	Followed by 100 hr. at 715 °C.
S	98	67	76
T	96	67	81
X	96	62	76
Z	92	41	54
Steel	25-Hour Nucleation at 670 °C.		
	No Further Treatment	Followed by 100 hr. at 670 °C.	Followed by 100 hr. at 715 °C.
S	87	65	68
T	88	65	70
X	89	55	66
Z	72	40	48

ing without the intermediate 715-degree Cent. (1320-degree Fahr.) quench. The similarity of the two sets of data (Table VI) indicated that this intermediate quench had no effect in influencing the subsequent rate of graphitization.

It must be assumed that the ferritic quench has no marked effect on rate of graphitization and, therefore, if any solution effect is accomplished, such solution does not promote subsequent graphitization unless it is accompanied with the strain induced by higher quenching temperatures.

INDUCING GRAPHITIZATION AT 715 DEGREES CENT.

From the results already reported, it appears evident that some condition or the presence of some substance promoting nucleation for graphitization plays an important role in determining how the steels will react on subcritical annealing. This concept is well supported by the results obtained from preannealing at 715 degrees Cent. (1320 degrees Fahr.) prior to the prolonged temper at 670 degrees Cent. (1240 degrees Fahr.), and by the fact that little graphitization can be obtained by a prolonged 715-degree Cent. treatment.



Fig. 16—Showing Relative Effect on Steel T of Nucleating 5 and 25 Hours at 670 Degrees Cent. (1240 Degrees Fahr.), After Austenitizing 1 Hour at 1000 Degrees Cent. (1830 Degrees Fahr.) and Water Quenching, Prior to Tempering for 100 Hours at 715 Degrees Cent. (1320 Degrees Fahr.). Quenched. $\times 7$. (A) Tempered 5 hours at 670 degrees Cent. (1240 degrees Fahr.) prior to 100 hours at 715 degrees Cent. (1320 degrees Fahr.) in covered lead. (B) Tempered 25 hours at 670 degrees Cent. (1240 degrees Fahr.) prior to 100 hours at 715 degrees Cent. (1320 degrees Fahr.) in covered lead.

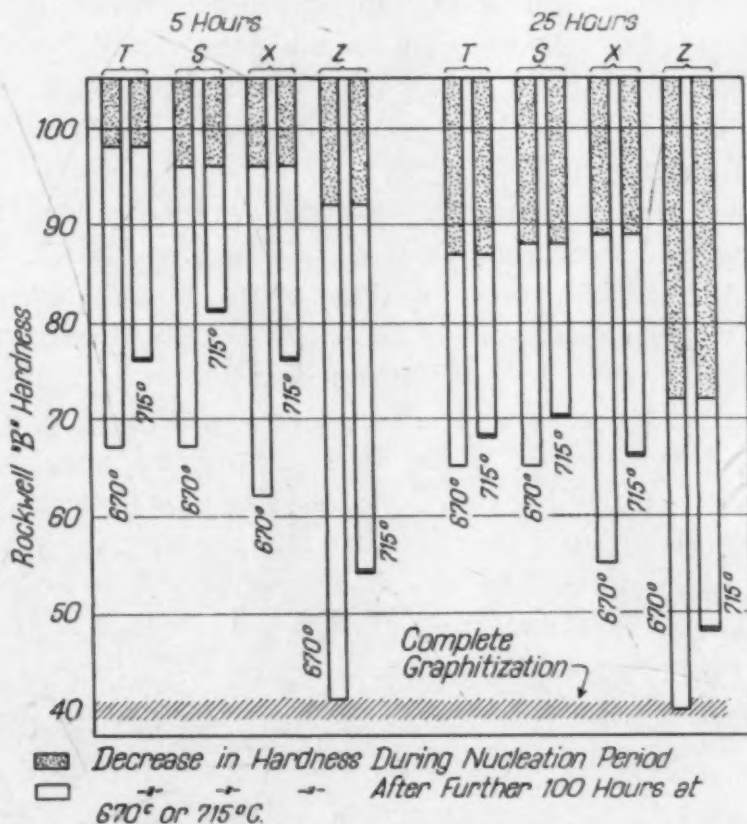


Fig. 17—Diagrammatic Illustration of the Graphite Nucleation Effect, Obtained by Tempering Water-Quenched Steels at 670 Degrees Cent. (1240 Degrees Fahr.), and Its relation to Subsequent Prolonged Tempering at 715 Degrees Cent. (1320 Degrees Fahr.)

At 715 degrees Cent. (1320 degrees Fahr.), the conditions for nucleation do not exist and such treatment apparently tends to destroy or render inoperative nucleation which would promote graphitization on tempering at lower temperatures.

The following interesting question therefore arises: If graphite nucleation is induced at, say 670 degrees Cent. (1240 degrees Fahr.), will the graphitization proceed on tempering just below the critical [715 degrees Cent. (1320 degrees Fahr.)]?

In this study four unstable steels, S, T, X, and Z, were subjected to graphite nucleation at 670 degrees Cent. (1240 degrees Fahr.) for periods of 5 and 25 hours. One set of the samples from both the 5-hour and 25-hour pretemper was subsequently tempered for 100 hours at 715 degrees Cent. (1320 degrees Fahr.) and another set was tempered for a further 100 hours at 670 degrees Cent. (1240 degrees Fahr.). All tempering was conducted in covered lead. Table VII shows the Vickers hardness numbers obtained.

Metallographic examination confirmed the indications given by hardness data, that all four steels graphitized as a result of the pretemper treatment but that the samples subjected for 25 hours had a larger number of graphite nuclei than those treated for 5 hours. This fact is evident from the unetched macrosection at 7 diameters (Fig. 16) taken from steel T.

The data demonstrated that although graphitization will not occur directly at 715 degrees Cent. (1320 degrees Fahr.), the dissociation of the carbides can be effected at this temperature so long as graphite nuclei are previously made available by some pretreatment at a lower or graphitizing temperature. The rate of graphitization is markedly less than that obtained at nucleating temperatures.

The bar graph in Fig. 17 has been constructed in an endeavor to indicate more clearly the results obtained by this nucleating treatment. From this figure it may be noted that the greater the number of graphite nuclei formed by the nucleating period at 670 degrees Cent. (1240 degrees Fahr.), the greater the drop in hardness on subsequent tempering at 715 degrees Cent. (1320 degrees Fahr.). For the 25-hour nucleating period the hardness values are from 8 to 12 points lower than for the corresponding treatment after a 5-hour nucleation.

It is interesting to note that there is a greater difference in tendency to graphitize at the two temperatures when the nucleating period is 5 hours than when 25 hours. This is probably due to the fact that

graphitization can take place at 715 degrees Cent. (1320 degrees Fahr.) provided graphite nuclei are present.

Since for the 25-hour period (Fig. 16) there are more nuclei than for the 5-hour period at 670 degrees Cent. (1240 degrees Fahr.), the extent of graphitizing at 715 degrees Cent. (1320 degrees Fahr.) approaches that of 670 degrees Cent. more closely.

Table VIII

Indicating Effect of Change of Environment (Atmosphere) for the Tempering Treatment at 670 Degrees Cent. for 125 Hours. Samples were tempered in both the normalized and water-quenched state in a lead bath protected from oxidation and in an oxygen-bearing lead bath. The hardness values are Rockwell "B"

Steel	—Water-Quenched from 1000 °C.—		—Normalized from 1000 °C.—	
	Covered Pb.	Uncovered Pb.	Covered Pb.	Uncovered Pb.
H	93	79	94	94
I	91	91	92	92
J	92	91	94	94
K	92	83	94	93
L	92	89	94	93
M	91	69	92	84
N	91	73	93	92
O	90	89	91	91
P	90	89	92	92
R	92	90	92	93
S	65	50	92	92
T	65	61	92	89
U	73	64	92	92
V	65	60	92	91
W	48	43	92	91
X	55	40	91	89
Y	46	38	87	62
Z	40	34	87	86

EFFECT OF ATMOSPHERE ON GRAPHITIZATION

Previous references (4), (6) have drawn attention to the fact that the presence of oxygen in the medium surrounding the steel specimens during the progress of annealing and during tempering may promote or accelerate the rate of graphitization at subcritical temperatures. Thus it is evident that atmospheric oxygen diffused from the uncovered lead baths into the steel, thereby reducing the apparent stability of the cementite.

Furthermore, later results show that the increase in rate of dissociation is dependent also on the pretreatment received by the steel. Thus, the development of susceptibility to graphitization in an oxygen-bearing tempering bath was most marked in steels which had been quenched from the highest temperature (1000 degrees Cent.), whereas normalized steels, with few exceptions, were not materially affected.

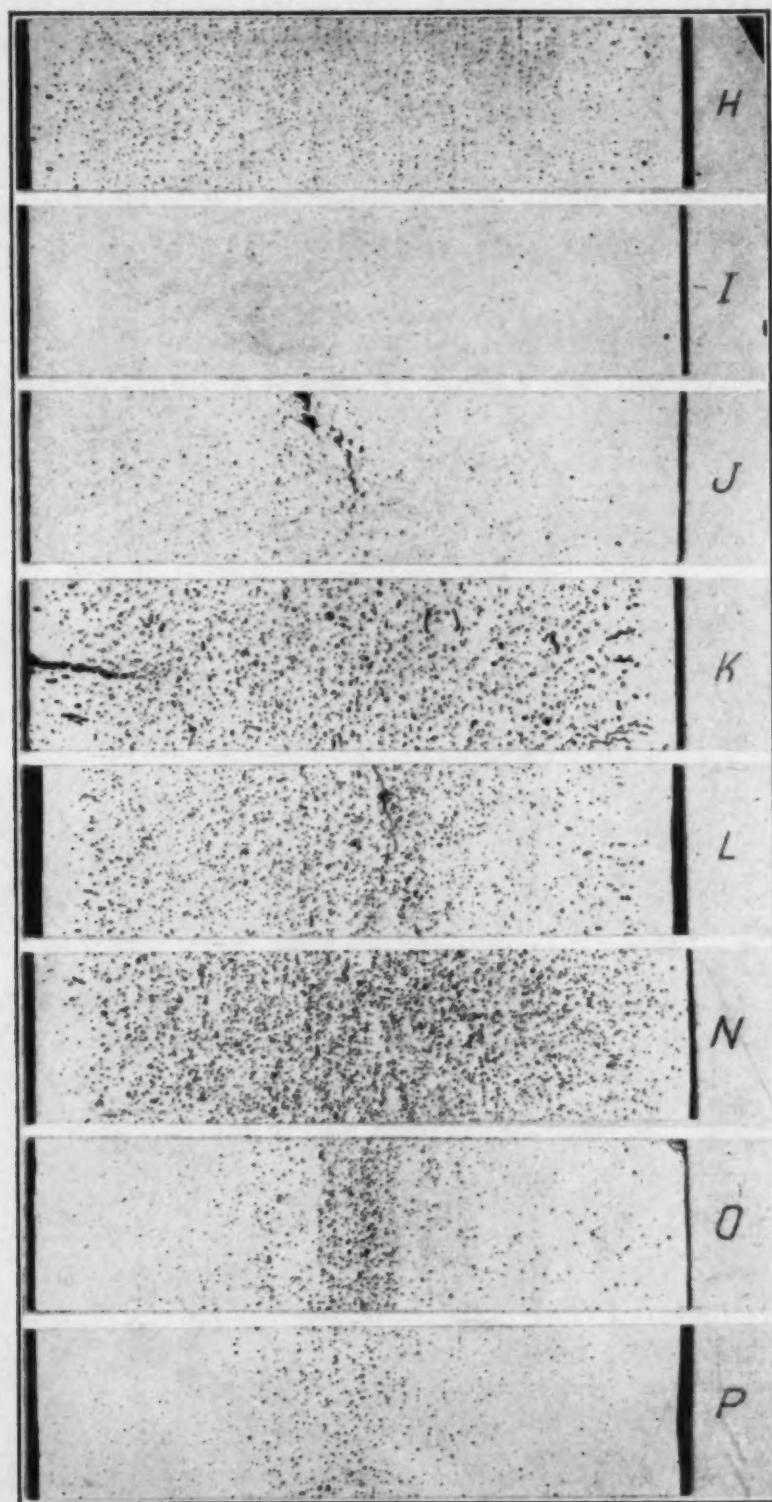


Fig. 18—Illustrating Important Role of Environment in Sub-critical Graphitization. The samples were austenitized by annealing 1 hour at 1000 degrees Cent. (1830 degrees Fahr.) in pot packed with charcoal and then water-quenched. They were then tempered for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) in lead bath unprotected from oxidation. Most of the stable steels exhibit graphitization. Unetched. $\times 7$.

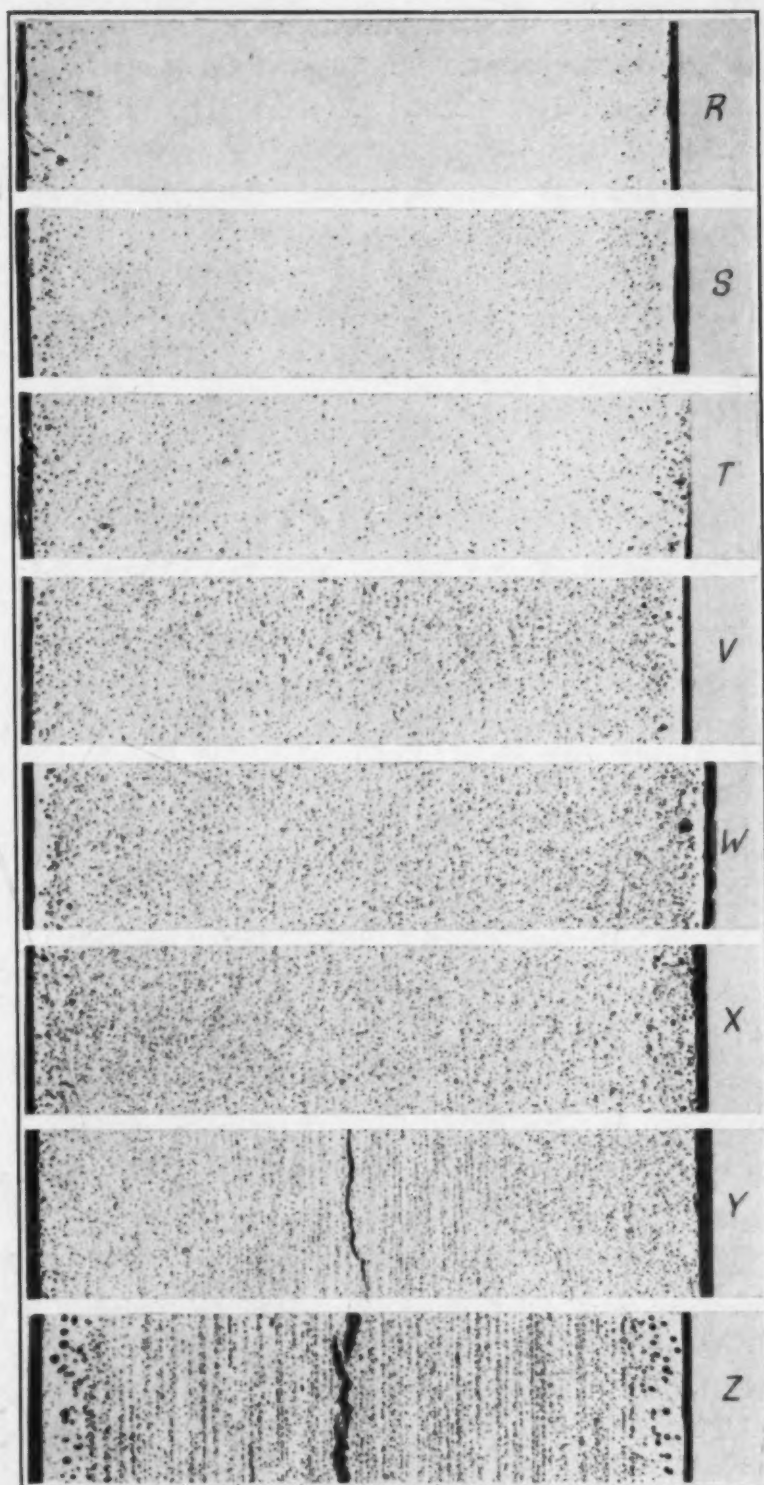


Fig. 18 (Cont.)—Illustrating Important Role of Environment in Subcritical Graphitization. The samples were austenitized by annealing 1 hour at 1000 degrees Cent. (1830 degrees Fahr.) in pot packed with charcoal and then water-quenched. They were then tempered for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) in lead bath unprotected from oxidation. Most of the stable steels exhibit graphitization. Unetched. $\times 7$.

After 125 hours in a lead bath at 670 degrees Cent. (1240 degrees Fahr.) with the surface unprotected from oxidation, initially clean steel surfaces were covered with an oxide scale. With the use of a covered lead bath the specimens were always clean and free from surface oxidation even after 600 hours' tempering. The absence of surface decarburization also confirmed the effectiveness of the charcoal covering used to prevent oxidation.

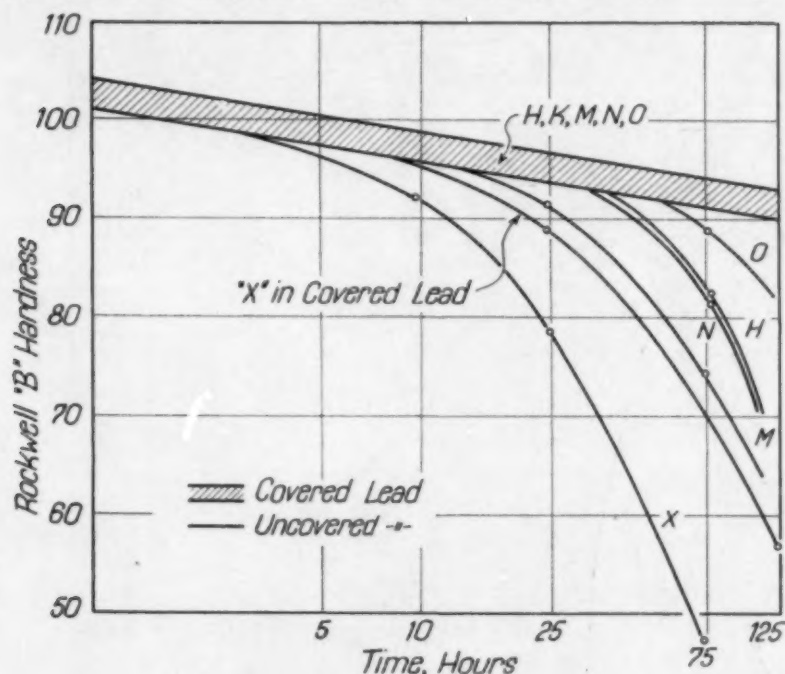


Fig. 19—Illustrating the Difference Obtained from Tests Conducted in Covered and Uncovered Lead on Rate of Graphitization as Indicated by Change in Hardness. The five stable steels (H, K, M, N, and O resistant in protected lead bath) showed marked graphitization on tempering at 670 degrees Cent. (1240 degrees Fahr.) beyond 25 hours in uncovered lead. The unstable steel X exhibits increased rate of carbide dissociation. The pretreatment consisted in water quenching for 1 hour at 1000 degrees Cent. (1830 degrees Fahr.) followed by 1-hour stress relief at 200 degrees Cent. (390 degrees Fahr.).

A complete picture of the role of oxygen during tempering at 670 degrees Cent. (1240 degrees Fahr.) for 125 hours in relation to water-quenched and normalized specimens is presented in Table VIII. After water quenching and tempering in covered lead, the stable steels (H and R) do not exhibit any carbide dissociation, whereas the unstable steels (S and Z) do show a drop in hardness from 90 Rockwell "B" down to values varying from 40 to 73. On tempering in uncovered lead many of the otherwise stable steels graphitize markedly as was indicated by the decrease in hardness. Macro- and micrographic examination demonstrated that all these steels (H and

R) graphitized to some extent. The stable steels most noticeably affected are M, H, N, K, O, and L (see Fig. 18 at 7 diameters) and of these, M, H, O, and N are higher in metallic aluminum than any of the other steels in this series. Steels K and L, although low in aluminum, were both treated with calcium silicide and steel K was higher in silicon (0.45 per cent) than any of the other steels. Steels I, R, and J appeared to have the lowest graphite content, as judged by the microstructure, and of these, R, and J were essentially free from metallic aluminum.

These observations appear to confirm the suggestion that the presence of oxygen (penetrating the steel by the normal process of diffusion), resulting in the formation of alumina and of calcium and silicon oxides, presents effective nuclei for carbide dissociation. It has been emphasized previously (4), that these oxidation products must be present in a certain physical state, as yet undefined, if the promotion of graphitization is to be profound.

Increase in degree of graphitization can also be noted from the hardness values (Table VIII) of the unstable steels when the tempering is conducted in an uncovered lead bath.

In Fig. 19, the traces for six of the steels (H, K, M, N, O, and X) correlating hardness with time at 670 degrees Cent. (1240 degrees Fahr.), for both protected and uncovered lead, clearly indicate the comparative softening rates.

Experimental studies were also made to ascertain if the uncovered lead tempering treatment was effective in graphitizing normalized steels. It will be recalled that all steels were relatively stable when tempered at 670 degrees Cent. (1240 degrees Fahr.) in covered lead after normalizing (hardness values Table VIII), and it was stated that steels Y and Z alone showed evidence, under the microscope, of graphitization. Indeed, normalizing appears to have a most effective stabilizing influence.

From the last column in Table VIII, it may be noted that the use of uncovered lead in tempering has little effect on the general reaction. Steel Y, however, still persists in softening markedly and steel M, usually regarded as stable, exhibits noteworthy softening characteristics. These two steels, together with S, were the most abnormal and the finest grained of any in the series, when subjected to the McQuaid-Ehn test.

The general results may indicate that either oxygen does not diffuse so readily at 670 degrees Cent. (1240 degrees Fahr.) unless

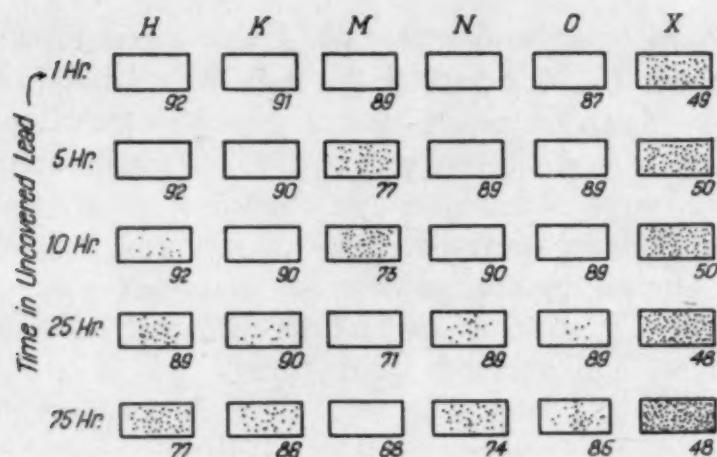


Fig. 20—Diagrammatic Indication of the Amount, Size, and Distribution of the Graphite Particles as Viewed Under the Microscope at 100 Diameters. The samples were water-quenched from 1000 degrees Cent. (1830 degrees Fahr.), pretempered from 1 to 75 hours at 670 degrees Cent. (1240 degrees Fahr.) in oxygen-bearing lead, and finally tempered 125 hours at 670 degrees Cent. (1240 degrees Fahr.) in a charcoal-covered lead bath. The numbers indicate the Rockwell "B" hardness values after the final treatment. (Note: The two blank rectangles under steel M at 25 and 75 hours should show heavy graphite spots instead of being blank.)

Table IX

Indicating Effect of Graphite Nucleation in an Oxidized Lead Bath at 670 Degrees Cent. on Subsequent Graphitization in a Lead Bath at 670 Degrees Cent. Protected from Oxidation. The specimens were austenitized by annealing for 1 hour at 1000 degrees Cent. and then water-quenched

Steel	Pretreatment after Quenching from 1000°C.	Rockwell "B" Hardness after Tempering		Pretreatment after Quenching from 1000°C.	In Covered Lead +125 hrs. in covered lead (3)	Difference between Columns (2) and (3)
		In Uncovered Lead No further treatment (1)	+125 hrs. in covered lead (2)			
H	1 Hour in Uncovered Lead	104	92	1 Hour in Covered Lead	93	1
K		103	91		92	1
M		103	89		91	2
N		102	87		91	3
O		101	87		90	3
H	5 Hrs. in Uncovered Lead	100	92	5 Hrs. in Covered Lead	93	1
K		100	90		93	1
M		98	77*		91	14*
N		99	89		91	2
O		98	89		90	1
H	10 Hrs. in Uncovered Lead	96	92	10 Hrs. in Covered Lead	93	1
K		98	90		92	2
M		97	73*		91	18*
N		97	90		91	1
O		97	89		90	1
H	25 Hrs. in Uncovered Lead	96	89	25 Hrs. in Covered Lead	92	3
K		96	90		91	1
M		91**	71*		90	19*
N		95	89		90	1
O		94	89		89	0
H	75 Hrs. in Uncovered Lead	83**	77*	75 Hrs. in Covered Lead	92	15*
K		89**	86		90	4
M		74*	68*		89	21*
N		82**	74*		89	15*
O		89**	85		88	3

*Indicates marked graphitization.

**Hardness values indicate some graphitization.

the steel was previously strained by quenching, or that if oxygen does penetrate, graphitization fails to result on account of the absence of internal strains. Abnormal steels were apparently so constituted that slight oxygen pick-up may render the carbides unstable.

Incidentally, no difference in hardness was noticed on traverses across the sections of the tempered rod. This indicated that any oxy-

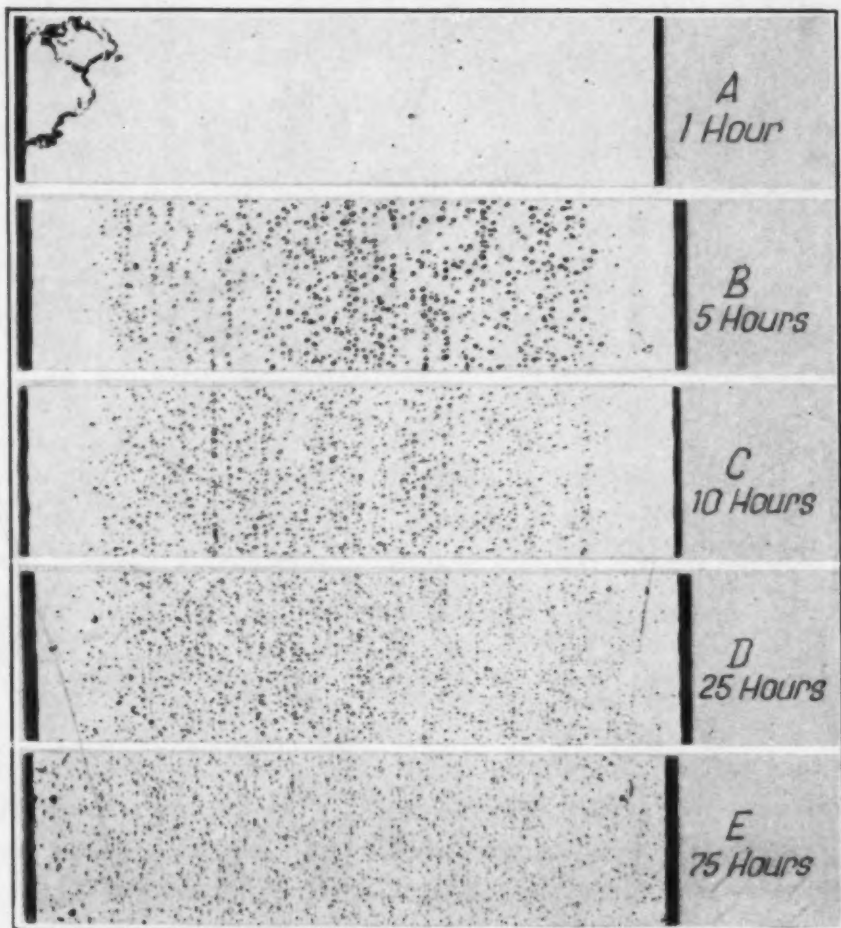


Fig. 21—Macrosections from the Normally Stable Steel M Quenched After 1 Hour at 1000 Degrees Cent. (1830 Degrees Fahr.), Nucleated with Graphite by Tempering in Oxidized Lead for 1, 5, 10, 25, or 75 Hours at 670 Degrees Cent. (1240 Degrees Fahr.), and Finally Tempered at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in a Lead Bath Protected from Oxidation. Unetched. $\times 7$.

gen effect which might be obtained was not gradually progressing inward from the peripheral parts of the sample.

Finally, we turn to a study of the effect of time of tempering in uncovered lead on subsequent graphitization, in a protected lead bath at 670 degrees Cent. (1240 degrees Fahr.). These experiments

relate to action of oxygen, diffused into the steel, on rendering the steels more susceptible to graphitization.

Five of the normally stable steels (H, K, M, N, and O) and the readily graphitizable steel X were held for 1, 5, 10, 25, or 75 hours in uncovered lead at 670 degrees Cent. (1240 degrees Fahr.) and then removed to the protected lead bath and held for 125 hours at

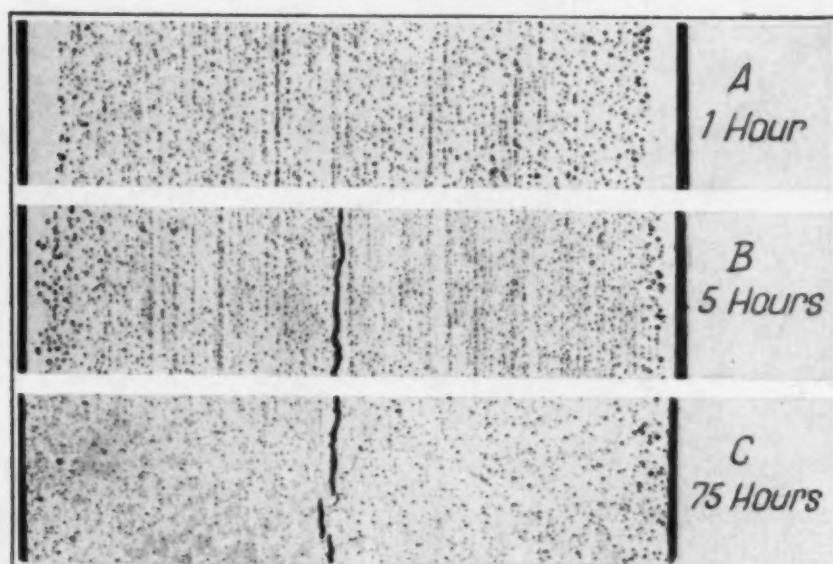


Fig. 22—Macrosections from the Unstable Steel X Quenched After 1 Hour at 1000 Degrees Cent. (1830 Degrees Fahr.), Nucleated with Graphite by Tempering in Oxidized Lead for 1, 5, or 75 Hours at 670 Degrees Cent. (1240 Degrees Fahr.), and Finally Tempered at 670 Degrees Cent. (1240 Degrees Fahr.) for 125 Hours in a Lead Bath Protected from Oxidation. Unetched. $\times 7$.

670 degrees Cent. (1240 degrees Fahr.). All samples were prior-quenched from 1 hour at 1000 degrees Cent. (1830 degrees Fahr.). By this procedure the graphite nuclei originating in the uncovered lead treatment could be made to grow to visible size with no probable increase in nuclei number.

The selection of the steels was based on oxygen susceptibility as indicated by previous tests and on the basis of aluminum content (see Table I).

From specimens $1\frac{1}{2}$ inches long, quenched from 1000 degrees Cent. (1830 degrees Fahr.), about $\frac{1}{4}$ inch was cut and discarded. This procedure provided a "clean end" surface from which to measure any penetration effects, dependent on subsequent heat treatment. Separate specimens were then tempered for 1 to 75 hours in uncovered lead, and sections cut, remote from the "clean end," for hardness determinations. The remainder of the 30 specimens were then

held for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) in covered lead, and microscopic examinations were made from the "clean end" inward on a longitudinal section through the center of the bar.

In Table IX will be found the complete set of hardness data in terms of Rockwell B, for the normally stable series of steels. In this table have also been included the hardness values obtained after tempering in covered lead, omitting the 1 to 75-hour pretreatment in uncovered lead. The last column gives a figure representing the amount of increased softening, and therefore of graphitization, resulting from the pretreatment under oxidizing conditions.

One-hour pretreatment in unprotected lead has little effect on any of the steels; however, 5, 10, and 15 hours' pretreatment has a profound effect on graphite nucleation in steel M, while the other steels in the series are affected only slightly. With a 75-hour pretreatment, steels H and N are also markedly affected and steels K and O exhibit some increased softening.

Sections of all the samples were examined under the microscope and possibly a more clearly summarized statement of their behavior is shown in the diagrammatic reproduction of Fig. 20. In this figure an attempt has been made to illustrate the appearance of the polished sections, when viewed under the microscope at about 100 diameters, with respect to size, number, and distribution of the graphite phase. Evidently the time at temperature (670 degrees Cent.), under oxidizing conditions, required to initiate graphite nucleation is different in the different steels, and the longer the period of treatment beyond this stage, the greater the number of nuclei and the finer the distribution of the graphite particles. This is similarly true of steel X, which graphitizes without the pretreatment of nucleation.

In order to demonstrate this striking picture with actual metallographic records, a series of macrosections have been reproduced at 7 diameters (Fig. 21) from the various treatments given to steel H. With the exception of the "rim effect", which is not dependent on decarburization, the illustrations are self-explanatory. Similar characteristics are shown for the normally graphitizable steel X (Fig. 22). Oxygen diffusion evidently increased nucleation and as the time of nucleation progressively increases, the banding effect is gradually obscured. This characteristic may be noted also in Fig. 21, relating to steel M.

The results obtained from these experimental studies indicate

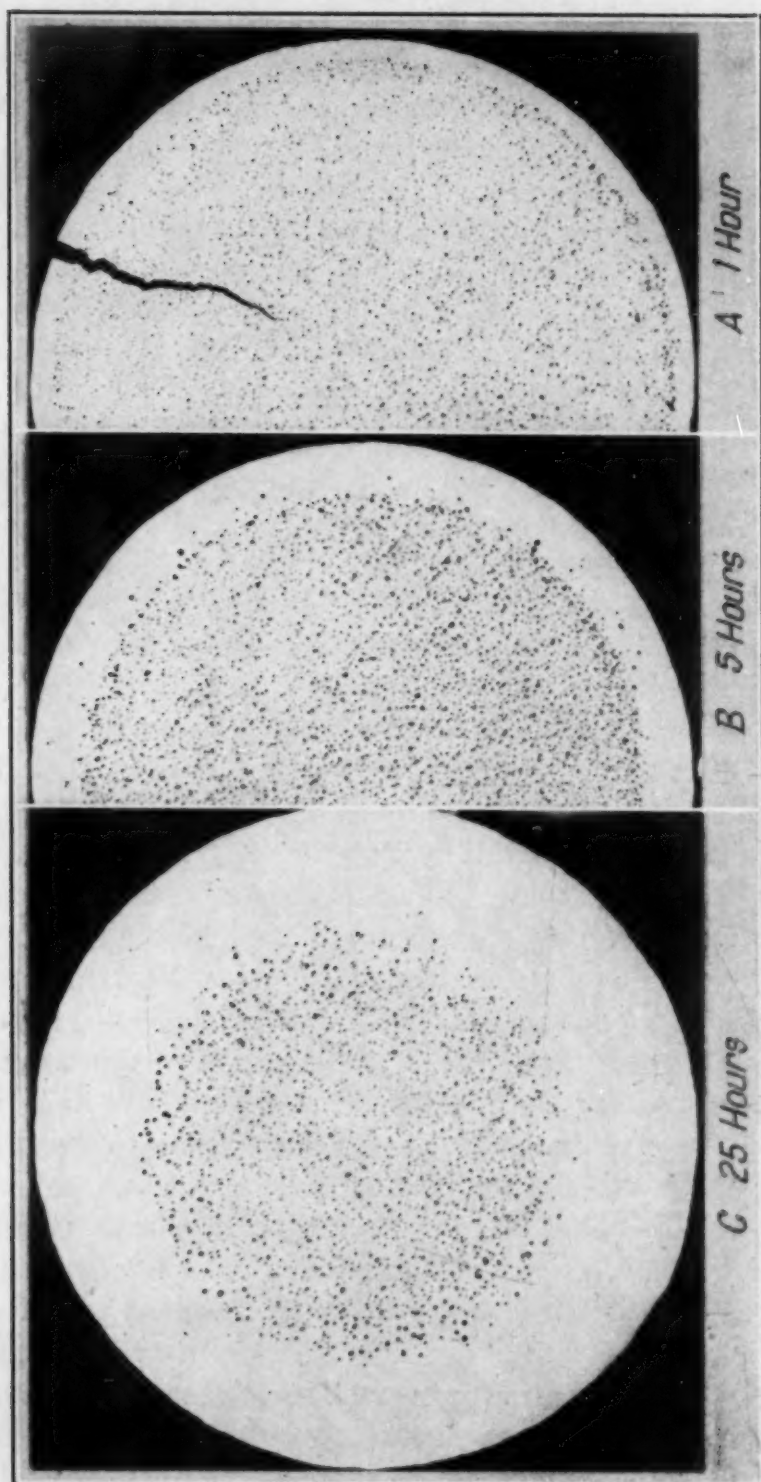


Fig. 23—Macrosections from the Unstable Steel T Quenched After Austenitizing 1, 5, and 25 Hours at 1000 Degrees Cent. (1830 Degrees Fahr.) in Pots Filled with Charcoal, and Graphitized for 125 Hours at 670 Degrees Cent. (1240 Degrees Fahr.) in a Lead Bath Protected from Oxidation. Unetched. $\times 7$.

that the degree of metastability of the cementite, and hence the susceptibility of graphite formation, may be dependent on mill practice, in terms of amount of minor element content such as aluminum and silicon, and melting or fabrication conditions which may promote internal oxidation of the steel. This oxidation of elements increasing metastability may occur while the steel is in the molten state, or in the soaking pit, reheating or annealing furnaces.

There is some support for this viewpoint in the discussion of Green's (7) paper on black fractures wherein J. A. Mathews stated that graphitization is due to bad mill practice, namely excessively high temperatures on the first cogging. The experimental data have already shown that less than 5 hours' exposure (as with steel M) to oxidizing conditions at 670 degrees Cent. (1240 degrees Fahr.) may provide the nuclei necessary to convert the carbides to elementary graphite and iron.

It is interesting to point out that the steels which were readily graphitizable in lead protected from oxidation were supplied by two steel companies, while the steels which have been considered as stable were shipped by two other steel companies. Furthermore, steel M, which has been classed as a stable steel, but which graphitized readily under an oxidizing environment, was manufactured about one year later than the rest of this series.

Some attention must be directed to the "rim effect" which has been mentioned earlier in the paper.

It was observed that a stable rim was obtained at the peripheral parts of a sample of steels which normally graphitized, if the specimen was protected from oxidation during annealing above the critical range, by surrounding it with charcoal granules. Similar results were obtained when the austenitizing was conducted in vacuum. The effects of vacuum treatment on graphitization of pure iron-carbon alloys containing a third element have been fully discussed (6).

The atmosphere surrounding a steel sample packed in charcoal is essentially carbon monoxide, and it would seem that this gas has the role of rendering ineffective the nuclei which cause the start of the breakdown of the metastable carbide.

Attention has already been drawn to examples of this rim effect and the results of a direct study of the problem are shown in Fig. 23. These three macrographs of the complete transverse sections of steel were obtained by austenitizing 1, 5, and 25 hours at 1000 degrees Cent. (1830 degrees Fahr.) in pots filled with charcoal, fol-

lowed by water quenching. The samples were then held for 125 hours at 670 degrees Cent. (1240 degrees Fahr.) in a lead bath covered with charcoal to prevent oxygen infiltration. Thus the thickness of the rim is a function of the austenitizing time in a carbon monoxide atmosphere.

With the eight graphitizable steels the depth of rim varied from 0.015 to 0.035 inch when held 1 hour at 1000 degrees Cent. (1830 degrees Fahr.). For steels T and X respectively, the depth of rim was 0.01 and 0.03 after 1 hour; 0.03 and 0.05 after 5 hours; and 0.07 and 0.095 after 25 hours austenitizing, followed by the 125-hour treatment at 670 degrees Cent. (1240 degrees Fahr.).

It was also noted that the rim effect was destroyed rapidly if the graphitizing anneal was carried out in a lead bath unprotected from oxidation. This statement is confirmed by reference to the illustration in Fig. 21.

A breakdown of the resistance to carbide dissociation also developed in a covered lead bath but the process was very slow. After 125 hours, some graphite particles could be discovered in the rim but the number was quite small.

The phenomenon was not, of course, associated with decarburization.

GENERAL CONSIDERATIONS

In previous researches, it has been pointed out that quenching from above the critical promotes graphitization upon subsequent tempering. However, this fails to explain why certain steels graphitize and others remain stable when subjected to identically the same treatment. For example, even after quenching from 1000 degrees Cent. (1830 degrees Fahr.) the stable steels (H to R) maintain their carbide stability upon tempering in covered lead. The unstable steels (S to Z) retain the general order of carbide instability regardless of quenching temperature, but show a progressive increase in graphitization as the quenching temperature is raised. The time at temperature prior to quenching appears to have relatively little importance.

On the other hand, graphite has been found after tempering steels (W, Y, and Z) which were coarsely pearlitic after slow furnace cooling. In this case, graphitization seems to have occurred without the accelerating action of quenching.

It has been pointed out, however, that reasons for the increased rate of graphitization on tempering as a function of austenitizing temperature may include such factors as high carbon content of martensite, reduced amount of free cementite, increased internal stress concomitant with the formation of microcracks, and probable increased solution of elements or compounds which may promote carbide instability. It was further suggested, however, that the observed changes in carbide instability would appear to be related more particularly to the increased strain associated with incipient microcracks and to increased solubility of some substances promoting graphitization, both dependent on high quenching temperature.

The concept of increased solubility of a phase promoting graphitization appears to receive direct support from the directional distribution of graphite formed after tempering. This phenomenon can be assumed to be associated with segregation and hence with localized solution of the disperse phase. This behavior has been clearly illustrated in the macrographic records presented for steel Y (Fig 7).

In the studies on supplementary pretreatments, the importance of the presence of martensite prior to tempering has been demonstrated. Thus oil quenching from 850 degrees Cent. (1560 degrees Fahr.) does not promote martensite and consequently does not cause graphitization on subcritical annealing. However, once martensitization is effected, repeated quenching from above the critical does not appear to modify appreciably the metastability of the carbide.

The results obtained as a result of cold deformation prior to tempering appear particularly interesting. Thus, while compressive stresses have little effect, tensile stresses may profoundly affect subsequent behavior on tempering unstable steels. Since the reaction $\text{Fe}_3\text{C} = 3\text{Fe} + \text{C}$ occurs with increase in volume, any resultant "loosening" of atomic or molecular structure of the matrix might be expected to facilitate dissociation of the unstable phase. However, the dissociation tendency of the carbides in the stable steels appeared to be unaffected by either type of mechanical strain.

Truly striking observations relate to the manner in which tendency to graphitization at 670 degrees Cent. (1240 degrees Fahr.) is profoundly reduced by pretempering at 715 degrees Cent. (1320 degrees Fahr.). This effect has been recorded in studies on the unstable steels and must be associated with some form of nucleation. As regards solubility effects, quenching of the ferritic steel from a

temperature just under the critical has been shown to have no importance.

While it has been shown that pretempering at 715 degrees Cent. (1320 degrees Fahr.) promotes the stability of the steels, evidence has been presented to demonstrate that as the amount of graphite nucleation is progressively increased by pretempering at 670 degrees Cent. (1240 degrees Fahr.), further tempering at 715 degrees Cent. (1320 degrees Fahr.) results in continuation of the processes of carbide dissociation.

The role of atmosphere has also received experimental consideration, and it has been shown that while the presence of oxygen in the environment used in tempering promotes graphitization, the increased susceptibility is dependent on prior austenitization followed by rapid cooling. The most noteworthy effect relates to the manner in which "stable steels" are rendered unstable when an oxidizing atmosphere is used during tempering. The results appear to lend strong support to the suggested role of aluminum (calcium and silicon) oxide, in promoting subcritical graphitization, when present in suitable form and suitable degree of dispersion. It is to be noted, however, that this phenomenon is exhibited only when such change in subcritical tempering environment is associated with rapid cooling following austenitization.

A further effect caused by the atmosphere obtaining during the process of austenitization has been pointed out. Substitution of carbon monoxide for air results in a progressive increase in thickness of a peripheral stabilized rim as the time of annealing is increased. Carbon monoxide has a definite reducing power and presumably has some reducing action on the substance or substances promoting graphitization. Indeed, when subsequent tempering is conducted in unprotected lead, the resistance to carbide dissociation developed in the rim of the sample is rapidly destroyed. If, however, the steel is protected from oxygen infiltration during tempering, the resistance of the carbides to dissociation persists.

One of the authors is happy to acknowledge the very able assistance rendered by Mr. Hugo R. Larson, a senior student in the department of metallurgy, during the preparation of this manuscript.

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DISCUSSION

Written Discussion: By H. A. Schwartz, manager of research, National Malleable and Steel Castings Co., Cleveland.

This writer is profoundly impressed with the scope and thoroughness of the subject paper. It is desired to point out some of the practical implications of this work.

There is a considerable volume of cast graphitizable steel made, having a carbon content not very far from 1 per cent, but rather high silicon contents, generally over 1 per cent. It was at first thought by this writer, when developing this product, that there would be no point in a high temperature treatment resembling the first stage of malleable annealing. It was almost immediately found that the graphitization of this material was much accelerated by heating to, say, 900 degrees Cent. (1650 degrees Fahr.), followed by fairly rapid cooling, though not by quenching, and subsequent heat treatment below the critical point.

This development in practice seems to be parallel with the authors' experimental work.

Although the present paper deals with somewhat hypereutectoid steels, and high pressure, high temperature pipe and fittings are generally moderately low in carbon, much of the authors' work throws light upon a problem which has vexed the power plant designer for some time.

It has been found that when these high temperature lines are assembled by welding, then after years of use at, say, 900 degrees Cent. (1650 degrees Fahr.) a very marked segregation of graphite is formed in those regions of the steel near the welded joint. These regions may be assumed to have had high temperature treatments and perhaps to still contain residual stresses. There is a suspicion that steels containing aluminum are prone to this difficulty, and the formation of Al_2O_3 , as pointed out by Austin and Fetzer, may be of distinct importance.

In this connection it may be interesting to report that a certain steel casting was found by the purchaser to have developed graphite in a central band. In this case there was no relation to any welding, none having been done in proximity to this area. Most of the casting extending inward from either surface was graphite-free, but a band, perhaps 0.5 cm. wide, extending somewhat irregularly down the axis of the cross section, contained graphite. Analysis in our laboratory showed approximately 0.003 per cent Al_2O_3 in the graphite-free zone and 0.007 per cent in the zone which had graphitized.

Written Discussion: By F. Eberle, research metallurgist, The Babcock & Wilcox Co., Barberton, Ohio.

This paper is another valuable contribution by the authors on the subject of graphitization in steels. It would appear from the presented experimental results that these high carbon steels can be induced to graphitize at certain subcritical temperatures irrespective of melting and deoxidation practice, if suitable conditions of pretreatment and environment are chosen. For, the authors state on pages 518 and 519, "On tempering in uncovered lead, many of the otherwise stable steels graphitize markedly as was indicated by the decrease in hardness. Macro- and micrographic examination demonstrated that all these steels (H and R) graphitized to some extent". However, on page 487, last sentence, it is said that "With the exception of steels I and R, even the so-called stable steels exhibited some graphitization on prolonged tempering under oxidizing conditions in uncovered lead". It would be quite important to know if the authors succeeded in graphitizing straight silicon-killed steel which has received no addition of aluminum or of any other similarly powerful deoxidizer. It is to be regretted that the graphitizing experiments with the so-called stable steels in uncovered lead were carried out for the relatively short time of only 125 hours. If the tempering time had been extended to 600 hours, the graphitizing tendency of the so-called stable steels would have been established with greater certainty.

In view of the practical value of a better understanding of the phenomenon of graphitization in steels, it is also to be regretted that the authors have not quantitatively determined the degree of graphitization in their more important samples. For instance, if the average number of graphite nodules per square inch cross section were known, together with the percentage of the graphitic carbon, the various factors influencing graphitization could be appraised more accurately. To determine the degree of graphitization of a sample by the hardness drop on tempering is only an approximation, particularly if graphitization is only slight. Furthermore, a clarification of the terminology employed in describing certain aspects of graphitization would lead to a better understanding and appreciation of results. Frequently, one encounters such terms as "promotion of graphitization", "acceleration of graphitization", "increased rate of graphitization", but, it is not always clear what these terms are supposed to mean with respect to number of nodules per unit area or volume plus total amount of graphite formed within a given time, or time elapsed until graphite makes its first appearance. This is not meant to be a criticism, but rather a suggestion to agree upon a method of describing certain aspects of the graphitizing process.

In view of the hypothesis advanced by the authors that increased solution of elements or compounds may be one of the factors responsible for the observed increased rate of graphitization on tempering as a function of austenitizing temperature, it would appear worthwhile if the authors would determine the influence of increasing austenitizing temperatures upon subcritical graphitization with a steel of eutectoid composition.

This writer expresses his sincere appreciation of the work done by Drs. Austin and Fetzer.

Written Discussion: By B. S. Norris, research metallurgist, York Corporation, York, Pa.

The authors are to be congratulated on the manner in which they have shown how various factors may promote graphitization in commercial high carbon steels. The paper is particularly interesting to me since the authors have been able to more completely explain the action of some of the factors with which I was concerned when connected with the graphitization program directed by Dr. Austin.

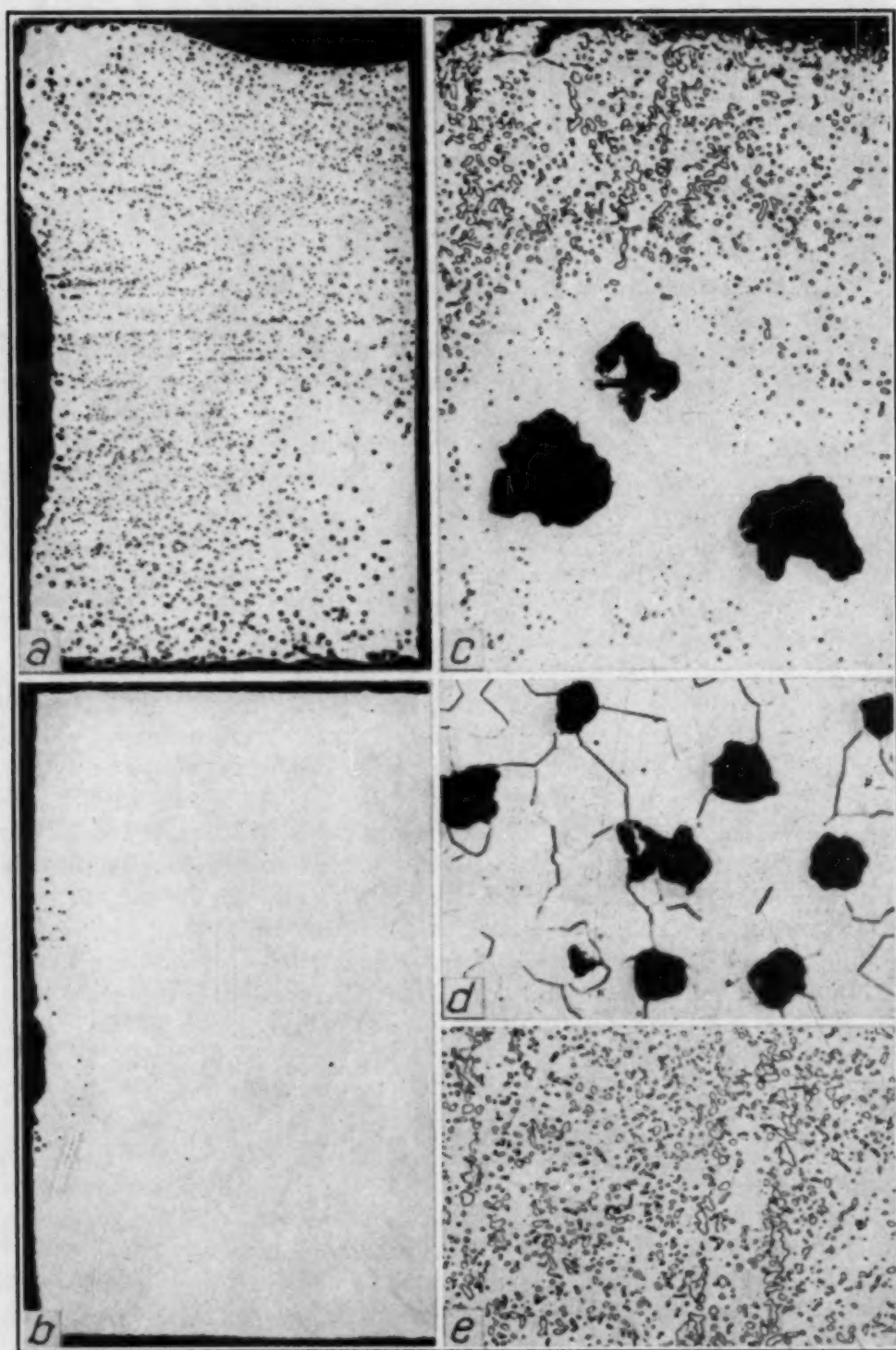
The presence of residual stresses appears to greatly increase the graphitization reaction provided the steel already has an urge to graphitize. According to Fig. 11 the closer an unstable steel (steel with an urge to graphitize) is stressed to its ultimate strength the greater is its graphitization. In other words, as the condition of the steel becomes more favorable for the formation of cracks the greater is the graphitization tendency. When a steel has a relatively high urge to graphitize, such as steel Z, much smaller stresses can initiate graphitization. This information raises the following question: Did the authors observe a greater cracking tendency among the unstable steels S to Z than in the stable steels H to R? Fig. 4 indicates that small cracks were probably present in steel Z after a water quench from a high temperature and a stress relieving anneal at 200 degrees Cent. (390 degrees Fahr.).

As a result of the authors' experience with cold-rolled samples it is concluded on page 527 that compressive forces exert little effect on graphitization. It is believed by me that if a steel has a great enough urge to graphitize, compressive forces can accelerate graphitization. However, their effect is probably much smaller than that exerted by tensile forces. In 1942¹ Dr. Austin and I presented data which showed that compressive forces would accelerate graphitization if the steel had a great enough urge to graphitize.

At this time I would like to recall several examples that were given in this earlier paper. A pure steel containing 1.14 per cent carbon and 0.37 per cent aluminum was used. The quantity of other chemical elements was small. This steel graphitized very easily when tempered in a covered lead bath at 630 degrees Cent. (1165 degrees Fahr.) following the formation of martensite by a water quench from 1000 degrees Cent. (1830 degrees Fahr.). However, when an "as-received" sample of the steel was heated for as long as 125 hours at 630 degrees Cent. (1165 degrees Fahr.) no graphite formed. The carbides were in the spheroidized condition when the steel was received.

When an "as-received" sample, containing Brinell ball impressions on a

¹Charles R. Austin and B. S. Norris, "Effects of Small Amounts of Alloying Elements On Graphitization of Pure Hypereutectoid Steels," *TRANSACTIONS, American Society for Metals*, Vol. 30, 1942, p. 425-457.

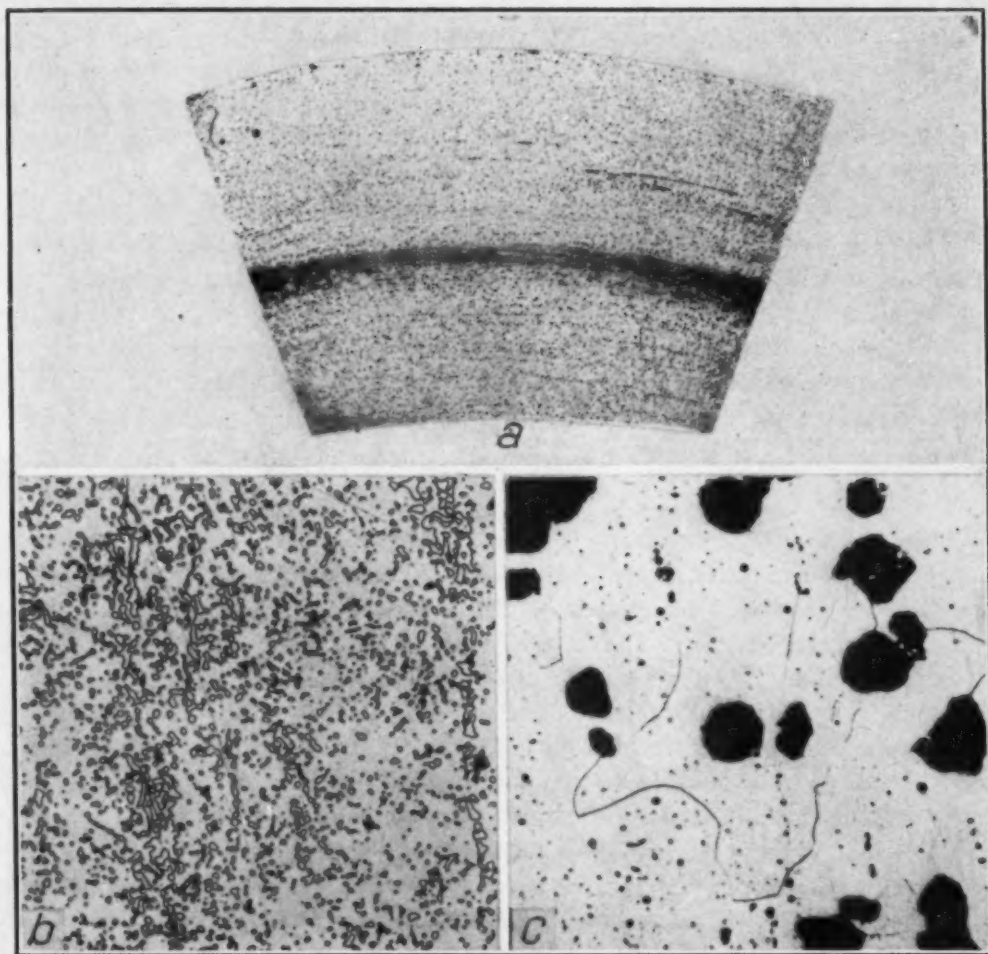


Figs. 24a to e—Longitudinal Sections of 0.37 Also 0.07 Aluminum Steels, Deformed With Brinell Impression in the As-Received Spheroidized Condition, and Then Tempered 125 Hours at 630 Degrees Cent.

0.37—Aluminum: a—One-Half Sample Showing Graphite Unetched and Two Brinell Impressions. $\times 7$; c—Etched Edge Near Impression. $\times 250$; d—Some Distance from Edge. $\times 250$; e—Much Further from Impression—Unstressed. $\times 250$. 0.07 Aluminum: b—Unetched Section Showing Graphitization Near Stencil Mark. $\times 7$.

freshly cut end and on the periphery, was heated in covered lead for 125 hours considerable graphite formed in certain regions of the sample. This is illustrated by the accompanying Figs. 24a to e.

Fig. 24a illustrates the appearance of an unetched longitudinal section which bisects the two Brinell impressions. The half of the sample omitted from Fig. 24a was practically free of graphite. The size and quantity of graphite formed appear to depend on the degree of cold working. These characteristics are



Figs. 25a to c—Sections Showing Effect of Strain on Graphitization on the 0.37 Aluminum Steel Tempered in As-Received, Spheroidized, But Strained Condition for 125 Hours at 630 Degrees Cent.

a—Etched Macrosection. $\times 5$. Showing Stable Neutral Axis; b—Etched Section on Neutral Axis. $\times 250$; c—Etched Section in Strained Area. $\times 250$.

well illustrated by Figs. 24c, d and e. Immediately under the impression made on the freshly cut end, left side of sample, the unchanged carbides are followed by very coarse graphitization (Fig. 24c), then by complete graphitization (Fig. 24d) and finally by an unchanged structure (Fig. 24e) at a location where the strain ceased to be effective.

In Fig. 24b graphite is shown near a stencil mark made on a pure steel containing 1.15 per cent carbon and 0.07 per cent aluminum. The graphite formed

when the "as-received" sample, containing spheroidized carbides, was heated for 125 hours at 630 degrees Cent. (1165 degrees Fahr.) in a covered lead bath.

A piece of the $\frac{3}{8}$ -inch diameter bar, in the "as-received" spheroidized condition, of the pure 0.37 per cent aluminum steel was bent cold around a 2-inch diameter mandril. An arc was cut from the resulting circle and tempered for 125 hours at 630 degrees Cent. (1165 degrees Fahr.). Longitudinal sections were cut and examined for macro- and microstructure. The resulting structures are shown in Figs. 25a to c. In Fig. 25a the top side of the bar was in tension while the bottom side was in compression during the bending operation. Along the neutral axis of the bar practically no graphite formed (Fig. 25b). However, in the regions where tensile and compressive forces had acted almost complete graphitization occurred (Fig. 25c). In this case the compressive forces appear to have been as effective as the tensile forces.

On page 519 the authors mention that steels M (stable), S and Y (unstable) were the most abnormal and the finest grained of any in the series when subjected to the McQuaid-Ehn test. Have the authors made any tests to determine whether a prolonged heating in an oxidized lead bath altered the normality or the austenitic grain size of a steel?

Have the authors ever tempered these steels at 670 degrees Cent. (1240 degrees Fahr.) after martensite had been formed by an isothermal quench?

Authors' Reply

The authors are appreciative of the comments made in the discussion of the paper. Dr. Schwartz's comments add to the information contained in the paper and emphasize some phases of the practical importance of the subject of graphitization on steels, particularly in power plant application. It is gratifying to note, from an examination of the recent literature on graphitization which has emerged as a result of failure in power plant lines, that the findings of the present authors and former colleagues, on high carbon steels, are directly applicable to the low carbon steels.

As regards Fig. 11, the meaning is clear, we believe, when the abscissa is made to read "tensile stress" instead of "tensile strength".

The comments of Dr. Eberle are interesting and they clearly demonstrate that he has given careful thought to an analysis of the factors which may promote graphitization in the type of failures already referred to. It should be pointed out, however, that this concluding paper, in a series of six published researches, was completed before attention was focused so emphatically on subcritical graphitization in steels under high temperature service conditions. Consequently, attention was directed toward ascertaining the factors promoting carbide dissociation, and the wide field relating to variation in deoxidizing methods in steel-making practice was not investigated. Nevertheless, the published researches have emphasized the vast experimental data necessary to give a general survey of the subject.

No doubt, now that the engineering importance of the subject is being recognized, many other investigators will give more thorough attention to the several factors which the researchers have shown to be relevant in the study of this subject.

Dr. Eberle appears to be particularly interested in graphite nodules and nucleation and correctly emphasizes the importance of this concept. While we have given some attention in the paper to relative amount or number of nuclei, Dr. Eberle will no doubt realize the experimental difficulties in stating precisely the number of nuclei at any given instant. Anyone who has attempted to follow the simple mechanism of spheroidization in steels, in the earliest stages immediately following martensite decomposition, can readily understand the difficulty encountered here in defining the absolute number of carbide particles in any field of the microscope.

We believe that Dr. Eberle has a very interesting suggestion regarding the study of the role of austenitizing temperature in steels which are as nearly exactly eutectoid in composition as practicable.

We are pleased to have the discussion from Dr. B. S. Norris, a former associate, and cannot add much to his comments. No efforts have been made to determine the effect of prolonged heating under oxidizing conditions on the normality of the steels, nor have we studied the effect on graphitization of martensitization by isothermal decomposition. But, then, does martensite form as a product of constant temperature decomposition of austenite?

IMPROVED SENSITIVITY IN DOUBLE-EXPOSURE RADIOGRAPHY

BY JAMES RIGBEY

Abstract

In order to overcome the decreased sensitivity inherent in double-exposure radiographs, a return is made to the stereoscopic method of exposing two separate films. In order that precise measurements can be made of the shift of a flaw image to determine the vertical position of the flaw, the image of a lead marker is used as a reference point. The measurement may then be corrected for the known shift of the marker image and used as in the case of double-exposure radiography.

In experiments with cast aluminum and steel blocks up to 2 inches in thickness, it was found that the consequent increased possible error in measuring the image shift does not appreciably alter the accuracy with which the position of the flaw is determined. Sensitivity is improved from 4 to 1 per cent while errors in results remain, in most cases, much better than 10 per cent of the specimen thickness. Maximum possible error in the actual measurements is calculated at about 14 per cent. Tests with production specimens gave similar results.

DOUBLE-exposure radiography is the method in which one X-ray exposure is superimposed upon another on the same film after the X-ray tube has been moved a short horizontal distance. By this means, if the specimen has not also been moved, the images of any part of it, corresponding to the two exposures, are separated by an amount which depends solely upon the distance of that part from the film, and other geometrical factors which can be determined. Thus it is used to introduce the third dimension into industrial radiography (1).¹ In this, it is a substitute for stereoscopic radiography as used in medical work, with the difference that the vertical position of any particular point is computed from measurements instead of being observed in a stereoscopic viewing stand.

It is quite obvious that the sensitivity obtained in double-ex-

¹The figures appearing in parentheses pertain to the references appended to this paper.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, James Rigbey, is radiologist, Ford Motor Company of Canada, Windsor, Ontario, Canada. Manuscript received May 16, 1944.

posure radiography is inferior to that of ordinary single-exposure radiographs. In a previous paper (2) it was shown that the best sensitivity attained in this type of work under the conditions of this investigation is around 2 per cent for aluminum and 4 per cent for steel. It is the purpose of this paper to demonstrate a method of attaining the customary sensitivity of 1 to 2 per cent and to determine the effect of this method upon the accuracy of the work in which the double-exposure technique is used.

This method involves a return to the use of two films exposed in the customary manner for stereoscopic viewing. That is, each of the two films corresponds to a different position of the X-ray tube, so that the images on the films, of various parts of the specimen, are in relatively different positions depending upon their relative distances above the film. Hence we may say that the image of a flaw undergoes a shift on the second film relative to the first, the magnitude of which depends upon the shift of the X-ray tube, the vertical position of the flaw in the specimen, and the focal distance or S.F.D. In order to measure the shift of the flaw image, a lead marker is placed at a known distance from the film (usually on the cassette) and in exactly the same position relative to the specimen on each film. The best method is to attach it with Scotch tape to either cassette or specimen. Then the difference between the measurements on the two films of the distance between the images of the flaw and the marker, measured in the direction of the tube shift and corrected for the known shift of the marker image, is a measure of the flaw image shift. This can then be used in the usual manner to determine the vertical distance of the flaw above the film.

Since this work was begun, an improved method of image shift measurement has been described (3). This method utilizes a lead grid next to the film and is applied to gamma radiography. The lead grid technique, however, appears to be more applicable to some types of specimens than to others and involves a specially constructed cassette. It might be applicable, with modifications, to all branches of radiography.

Two methods of using this measured shift were employed here. The first was to multiply by the factor (marker-film distance/marker image shift) or S.F.D./tube shift. The second was to plot empirically the curve of which the above factor is the slope. This second method eliminates the error which is present in the first due to the assumption that the X-ray beam is parallel instead of conical,

while obviating the use of the precise theoretical formula which is more laborious. It has been found, however, that the error introduced by the above assumption is negligible for reasonable target-film distance/specimen-film distance ratios.

PRELIMINARY WORK

The fact, that in the second exposure the specimen must be in exactly the same position as before, introduces some difficulty in practice. This can be controlled by orienting the cassette in the same manner each time with respect to its support and the specimen relative to the cassette. One method which helps considerably, when available, is to utilize a light placed in the radiographic cone. In the work described here, the X-ray tube was provided with a lead shutter and a medical type cone with balsa wood at the lower end (for the purpose of comfortable contact with the patient's skin). Consequently it was possible to tape a small 110-volt light bulb near the lip of the shutter and power it from the terminals of the shutter motor. Holes were drilled in the balsa plug, at each corner and in the center, so that the light beams through the holes approximately outlined the X-ray beam and its center. Thus by changing cassettes before moving the tube, the orientation of the specimen can be repeated by reference to the light beams.

The light beam method was found to be very useful in indicating on the film the direction of the tube shift. Before positioning the tube for exposure, it was set at two positions on its tracks and the positions of the center light beam on the cassette marked with lead arrows. After processing, the images of these two markers were joined by a scratch in the emulsion which then indicated the direction of tube shift.

In order to measure the distance between the flaw and marker images, a celluloid square was used. Since such squares with a millimeter scale along one edge were unavailable at the time, a convenient one was made from X-ray film. A negative was first made by placing a celluloid rule on the film and giving it a second or so exposure with white light. A positive was then printed off this in the same manner and a solid right-angled triangle cut out with the scale image along one edge. Error in the scale, introduced through shrinkage of the emulsion, amounted to only about 0.2 per cent and was neglected. Lines were scratched in the emulsion

parallel to the scale edge and about 1 centimeter apart to assist in aligning the edge parallel to the scratch denoting the direction of tube shift. The use of the square is illustrated in the photograph in Fig. 1.



Fig. 1—Photograph Showing Use of the Celluloid Square in Determining the Distance Between the Flaw at "A" and the Marker "B" in the Direction Parallel to the Tube Shift.

Tube shifts of 30.5 and 61 centimeters (1 and 2 feet), and an S.F.D. of 114 centimeters (45 inches) were chosen for all the ensuing work. Working curves were made by placing lead markers on wooden blocks of various thicknesses up to 7 centimeters and making a double exposure in the usual manner. The distances between the two images of each marker were then plotted against their distances above the cassette including half the thickness of the markers. The resulting curves A and B are shown in Fig. 2 for 1 and 2-foot tube shifts using High Speed film. The intersection of the two curves with the vertical axis is, of course, the measure of the distance of the film below the surface of the cassette. Similar

curves were made for Noscreen film for use with light alloys and thinner steel specimens. The deviation of each of these empirical curves from the straight line of slope S.F.D./tube shift is due, of course, to the conical shape of the X-ray beam and shows clearly that, for an S.F.D. of 114 centimeters, the parallel beam assumption

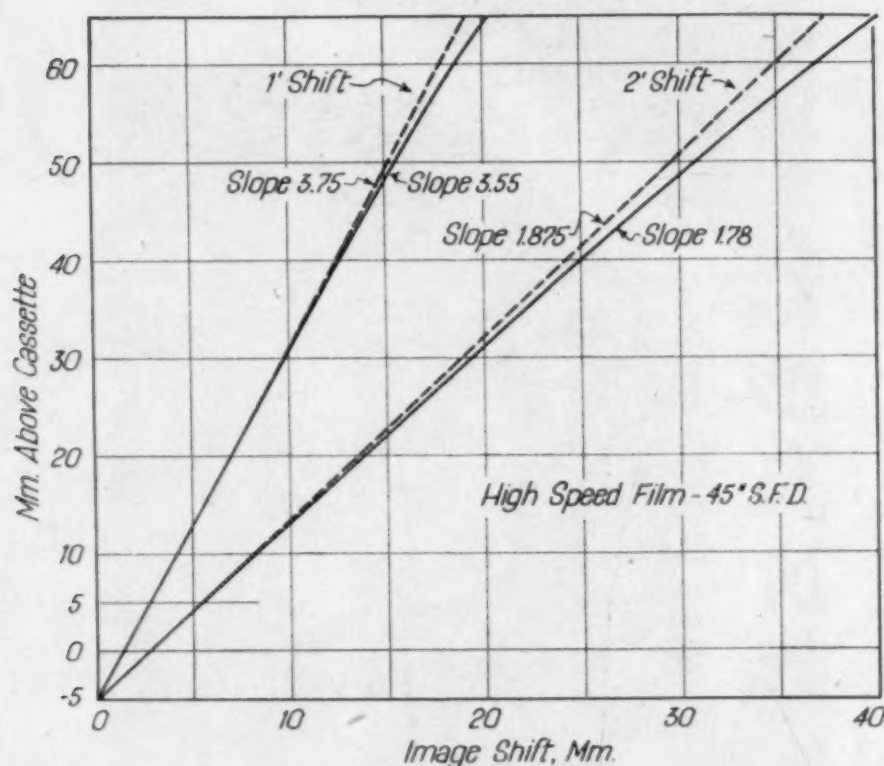


Fig. 2—Curves Showing 1- and 2-Foot Tube Shifts Using High Speed Film.

introduces appreciable error in the determination of flaw-film distances in excess of about 5 centimeters. On comparing the precise and approximate formulae (2), we see that this error may be measured in the form $FS^2/(T^2 + TS)$ where F is the S.F.D., T the tube shift and S the flaw image shift. Using this formula, we arrive at the dotted straight lines in Fig. 2 which are seen to possess the required slope.

Some care is required in using the measured flaw image shift, depending upon the position of the flaw relative to the reference marker. If, for example, the marker be on the cassette, it is best to use this quantity, uncorrected for marker image shift, with the factor S.F.D./tube shift, to get the distance of the flaw above the cassette. Using the graph, it will have to be increased by the image

shift of the marker to give the same quantity. If the marker is not on the cassette, it should be on top of the specimen so that it is known to be definitely above the flaw. This is because, if it be above, then the measured quantity will have to be treated as being negative since the image shift of the marker is greater than that of the flaw. To this negative measurement must then be added the positive quantity (marker image shift minus the image shift of a marker on the cassette) which may be obtained by reference to the graph. This is then ready for use with the factor. It is easier here, however, to use the graph since, for this, only the marker image shift need be added to the measured quantity.

EXPERIMENTAL PROCEDURE

The same test castings as were used in past experiments were again employed and have been described elsewhere (2). In brief these were, for work with aluminum, $\frac{1}{4}$, $\frac{3}{4}$ and $1\frac{1}{4}$ inches thick and were superimposed to form test specimens 1 and 2 inches thick (2.5 and 5.1 centimeters). Twelve holes 6.5 millimeters in diameter and varying in depth by about 0.8 millimeter were drilled in each. The point of the drill was 1.6 millimeters long and was not recorded in the measurements when the full diameter of the hole was visible on the film. For holes whose image diameter was less than 6.5 millimeters one-half the tapered tip was included in the recorded depth. Apart from these latter holes (all under 2 millimeters deep) the maximum possible error in measurement of hole depth or specimen thickness was ± 0.3 millimeter.

The steel specimens used were cast blocks $\frac{1}{2}$, 1 and $1\frac{1}{2}$ inches thick, superimposed to form specimens of 1 and 2 inches. These contained machined slots 0.3 centimeter wide by 1.2 centimeters and varying in depth from 0.25 to 2 millimeters (0.01 to 0.08 inches) in steps of 0.25 millimeter. Possible error ± 0.1 millimeter. In both types of specimens the holes were on top and at the interfaces of the superimposed castings.

The tube shifts were 61 and 30.5 centimeters for the 2.5 and 5.1-centimeter specimens respectively as recommended previously. The tube positions were both equally spaced each side of the specimen and also directly overhead and with the complete shift made to one side of the specimen. Either method was found to be practicable and consequently the technique described in reference 4 may be employed.

The tube used was an oil-cooled Standard "Flexray", 220-k.v.p. unit with focal spot rated at 2.4 millimeters by the dealer. Exposures were all made at 5 m.a., time being measured by means of a timer operating a motor-driven shutter. S.F.D. was 114 centimeters as before.

Noscreen film with 0.2-millimeter lead filter was used for work with aluminum while High Speed film with two calcium tungstate intensifying screens was used with the steel, utilizing copper shot for blocking purposes.

RESULTS

In working with aluminum, holes over 1.5 millimeter deep in the 2.5-centimeter specimen and over 5 millimeters deep in the 5.1-centimeter specimen showed sufficiently clearly to estimate the positions of their extremities. When measuring the shift for the center of the holes, the centers of the circular images were used. Steel specimens did not show the extremities of the holes sufficiently clearly for measurement. In this case, the distance measured was from the point of the marker image (usually an arrow) to a corner of the machined hole image. All calculations were made to determine the distance from the bottom of the specimen.

Combining the maximum possible errors in setting the tube positions, the S.F.D. and repositioning the specimen for the second exposure, we find them to be 1 or 2 per cent depending on the tube shift. With reasonable care in making measurements on the film, the maximum possible error from this source is about $\frac{1}{2}$ millimeter per measurement and about half this for the marker image shift taken from the graph. Thus we get the maximum possible error in image shift determinations to be 1 to $1\frac{1}{2}$ millimeters, depending on the method used. This gives a result of 8 to 12 per cent error taken as percentage of the specimen thickness when a 61-centimeter tube shift is used for a 2.5-centimeter specimen and so on as done here. Combining the above and taking the possible error in dimensions as measured by calipers and depth gage to be negligible, we find that, depending upon the method used, the maximum possible error is 9 to 14 per cent. In addition to this, the variable error when using the straight line slope factor must be considered in determining flaw-film distances in excess of 5 centimeters.

The errors in calculating the positions of the holes are listed in the following table as percentages of the specimen thickness. The

bracketed numbers are the average values neglecting the sign when both positive and negative errors are averaged.

A. Specimen: Aluminum, 2.5 centimeters

1. Exposure at 60 k.v.p.: 10 minutes.

Tube positions: 30.5 centimeters each side of specimen.

Holes and marker at top of specimen.

METHOD	TOP OF HOLE	BOTTOM OF HOLE	MIDDLE OF HOLE
Factor	-4 to -8 (6)	+2 to -10 (5)	+2 to -6 (3)
Graph	-2 to -6 (4)	+4 to -8 (6)	+4 to -4 (3)

Tops of holes 6.5 millimeters from top specimen. Marker on cassette.

Factor	+6 to 0 (4)	+14 to +2 (8)	+10 to 0 (6)
Graph	+8 to 0 (6)	+14 to +6 (8)	+12 to 0 (7)

2. Exposure at 90 k.v.p.: 2 minutes.

Tube positions: 30.5 centimeters each side of specimen.

Holes and marker at top of specimen.

Factor	+4 to -8 (4)	0 to -8 (4)	0 to -8 (3)
Graph	+6 to -8 (4)	0 to -8 (4)	+2 to -6 (2)

Tops of holes 6.5 millimeters below top specimen. Marker on specimen.

Factor	+8 to 0 (4)	+12 to -2 (5)	+8 to 0 (3)
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Graph same as factor.

3. Exposure at 120 k.v.p.: 2 minutes.

Tube positions: overhead and 61 centimeters to one side of specimen.

Holes and marker at top of specimen.

Factor	-2 to -10 (4)	+4 to -10 (6)	-2 to -8 (5)
Graph	+2 to -4 (2)	+4 to -8 (4)	0 to -6 (3)

Tops of holes 6.5 millimeters from top specimen. Marker on cassette.

Factor	0 to -10 (5)	+6 to -8 (5)	+4 to -12 (6)
Graph	0 to -10 (5)	+8 to -4 (4)	+6 to -10 (6)

B. Specimen: Aluminum, 5.1 centimeters.

1. Exposure at 100 k.v.p.: 10 minutes.

Tube positions: 15.2 centimeters each side of specimen.

Holes and marker at top of specimen.

Factor	+4 to 1 (2)	+10 to 4 (6)	+5 to -6 (2)
Graph	+4 to 1 (3)	+5 to 0 (2)	-2 to -11 (5)

Tops of holes 32 millimeters from top specimen. Marker on cassette.

METHOD	TOP OF HOLE	BOTTOM OF HOLE	MIDDLE OF HOLE
Factor	0 to -4 (3)	0 to -2 (1)	+6 to -11 (5)
Graph	-2 to -5 (4)	0 to -2 (1)	+3 to -12 (5)

2. Exposure same.

Tube positions: overhead and 30.5 centimeters to one side of specimen.
Holes and marker at top of specimen.

Factor	+8 to 0 (4)	+11 to +2 (4)	-+7 to -6 (4)
Graph	+2 to -4 (2)	+ 6 to -3 (2)	+1 to -8 (5)

Tops of holes 19 millimeters from top specimen. Marker on cassette.

Factor	+4 to -3 (4)	+8 to 2 (4)	+8 to -6 (4)
Graph	+1 to -7 (2)	+4 to -4 (2)	+5 to -7 (3)

C. Specimen: Steel, 2.5 centimeters.

Exposures at 120 k.v.p.: 2 minutes.

1. Tube positions: 30.5 centimeters each side of specimen.

Holes at top of specimen. Marker on cassette.

Factor	Graph
+4 to -2 (2)	0 to -6 (3)

Tops of holes 12 millimeters below top specimen. Marker on tray.

+4 to -4 (2)	+2 to -6 (3)
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2. Tube positions: 15 centimeters each side of specimen. (Note smaller tube shift.)

Holes and marker at top of specimen.

+12 to 0 (6)	+8 to -2 (3)
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Tops of holes 12 millimeters below top of specimen. Marker on tray.

+4 to -8 (4)	+4 to -10 (5)
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3. Repetition of 1 with specimen turned 90 degrees about vertical axis.

Holes and marker at top of specimen.

+8 to +2 (5)	+2 to -2 (2)
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Tops of holes 12 millimeters below top of specimen. Marker on tray.

+6 to -2 (4)	+2 to -2 (2)
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D. Specimen: Steel, 5.1 centimeters.

Exposures at 200 k.v.p.: 2 minutes.

Tube positions: 15 centimeters each side of specimen.

Marker on tray.

Holes at top of specimen. (2 radiographs.)

+9 to +1 (6)	+3 to -3 (2)
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Tops of holes 32 millimeters below top of specimen.

+2 to -4
(2)

+1 to -5
(3)

Tops of holes 26 millimeters below top of specimen.

+3 to -1
(2)

+1 to -3
(1)

From the above results it will be noted that the actual errors generally fall well within the calculated maximum experimental errors so that in these examples, at least, the calculated errors allow sufficiently for variation in contrast and definition. This is particularly noteworthy in the three examples using specimen A since, with a light metal, variation in technique has a marked effect upon the contrast (5).

Using the straight line slope as a multiplying factor, we see that the results coincide quite well with those obtained by using the graph, particularly when the holes are close to the film. They become slightly more positive when the 5-centimeter samples are used and in this case it might be more accurate to use the factor obtained by measuring the slope of the best straight line drawn through and among the points on the curve, i. e., 1.78 instead of 1.875 for the 61-centimeter tube shift and 3.55 instead of 3.75 for the 30.5-centimeter tube shift, as shown in Fig. 2.

In order to test the method with regular samples, two which contained several blow holes were radiographed in the above manner and then sectioned to expose the holes. The results were as follows:

Specimen: Steel Sprocket, 1.9 centimeters thick. (See Fig. 1.)

Exposure: 2 minutes at 100 k.v.p.; High Speed Film, Copper Shot Blocking.

Tube Positions: 30.5 centimeters each side of specimen.

Marker on Tray.

Hole	Depth Mm.	Distance of Center of Hole to Bottom Casting			Errors Per Cent of 1.9 Centimeters	
		Calculated		Measured Mm.		
		Graph	Factor			
		Mm.				
1	1	3	2.5	2.5 ± 0.5	+2.5	0
2	1	6.5	6.5	5.5 ± 0.5	+5	+5
3	1	1	1	2.5	-7.5	-7.5
4	1	5.5	5.5	5.5	0	0
5	1	3	2.5	3	0	-2.5
6	2	2	2	3 ± 0.5	+5	+5
7	2	5.5	5.5	6.5 ± 1.0	-5	-5
8	.	4	3.5	6.5	-12	-15

Specimen: Steel Bracket, 6 to 20 millimeters thick, 4 to 50 millimeters from cassette. (Fig. 3.)

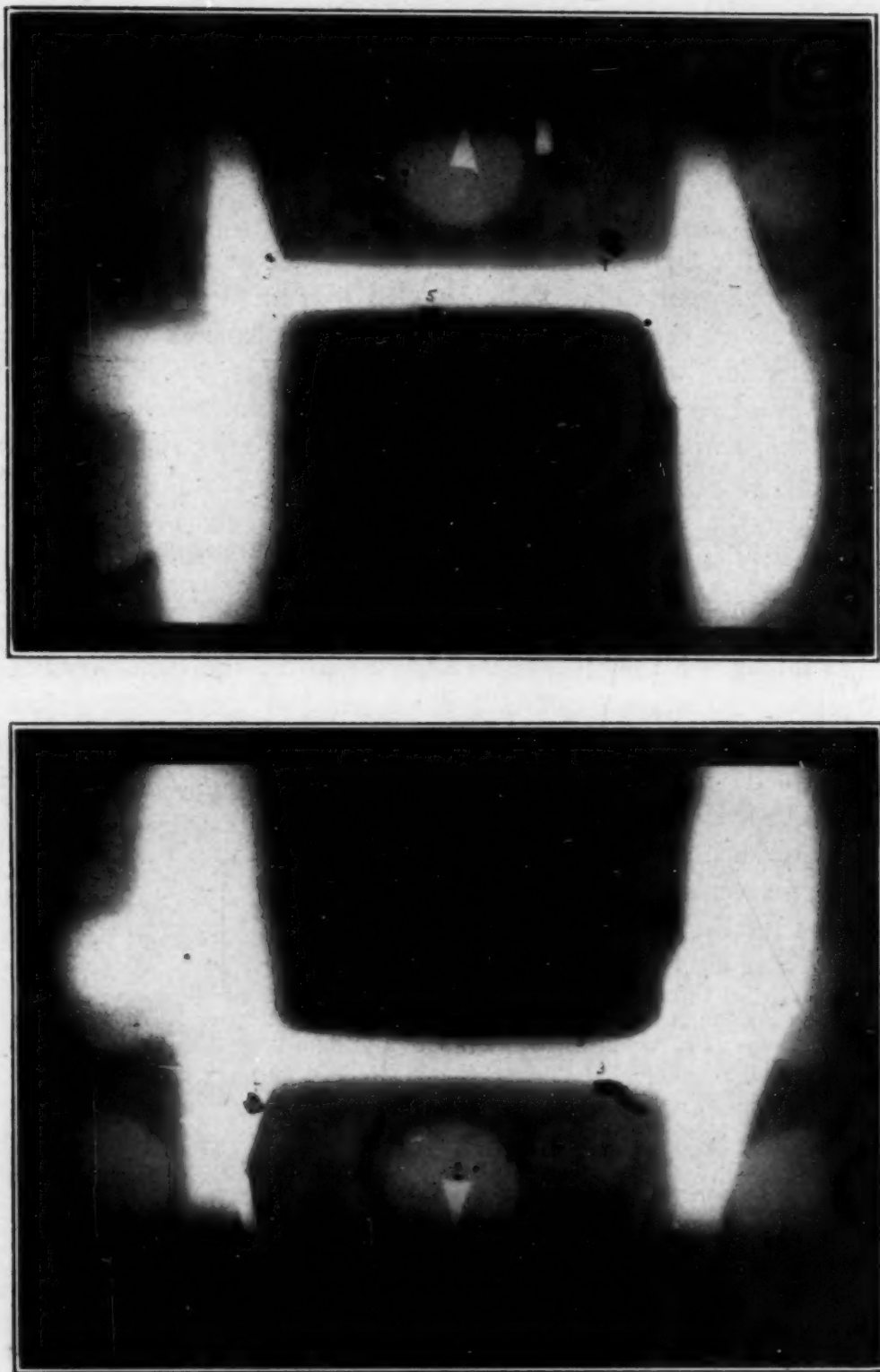


Fig. 3—Steel Bracket with Numbered Cavities Used to Test the Modified Double-Exposure or Stereoscopic Technique. Note How Consideration Must be Made of the Possibility of Losing a Flaw Image by Projecting it Under a Vertical Portion of the Specimen When Using Certain Tube Angulations.

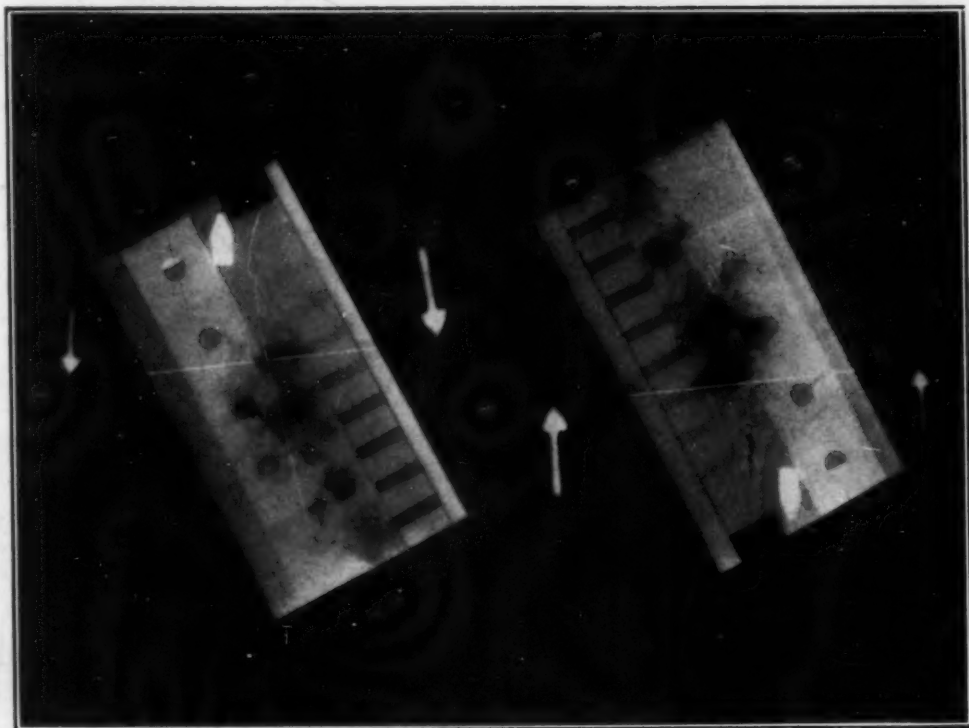
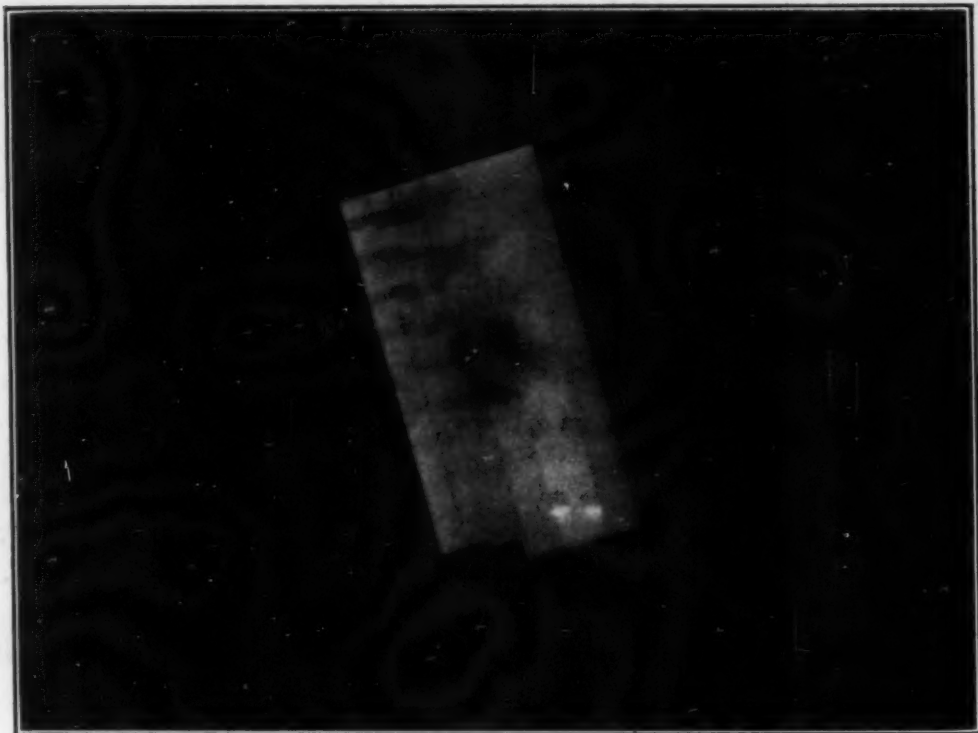


Fig. 4—Half-Inch Steel Block Showing Decreased Sensitivity and Confusion of Detail in Massive Porosity Resulting from the Use of the Customary Double-Exposure Technique (Upper). The Separated Exposures in the Lower Picture May, in Addition, Be Used in a Stereoscopic Viewing Stand If So Desired.

Exposure: 5 minutes at 200 k.v.p. Noscreen film, 0.2-millimeter lead filter.
 Tube Positions: 30.5 centimeters each side of specimen.
 Marker 2.5 centimeters from cassette.

Hole	Depth Mm.	Distance of Center of Holes to Cassette			Errors Per Cent of 2.5 Centimeters	
		Calculated		Measured Mm.		
		Graph	Factor			
		Mm.				
1.	1.5	5	4.5	5.5	-2	-4
2.	2	7	6.5	7	0	-2
3.	2	5	4.5	6	-4	-6
4.	4	6	5.5	6	0	-2
5.	4	54.5	56.5	55.5	-4	+4

On considering the irregular shapes of these holes, the errors are surprisingly small. The large errors in determining the position of hole No. 8 in the first sample were largely due to this source. The hole was elongated in an oblique direction and it was difficult to identify the same part of it in the two radiographs.

In general, it can be said that, under the conditions of this work, the return to the use of two films instead of a double exposure on one is justified by the improvement in sensitivity from 4 to about 1 per cent (Fig. 4). The decreased accuracy of the measurements appears to be practically negligible when reasonable care is taken. The chief drawback appears to be in the more complex calculations required in order to arrive at the simple measurement of image shift. However, in some circumstances these are simplified because they cancel out the correction for the distance between the film and the top of the cassette.

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1. Herbert R. Isenburger, "A Step Further in X-Ray," *American Machinist*, January 28, 1932, p. 133.
2. James Rigbey, "On the Location of Flaws by Double-Exposure Radiography," *TRANSACTIONS, American Society for Metals*, Vol. 31, September 1943, p. 599.
3. Benjamin B. Burbank, "Improved Stereoscopic Radiography," *Industrial Radiography*, Vol. II, No. 2, 1943, p. 20.
4. Michael Joseph, "Measuring the Depth of a Defect," *Industrial Radiography*, I, No. 4, 1943, p. 29.
5. R. C. Woods and V. C. Cetrone, "Radiography of Light Alloys," *Industrial Radiography*, II, No. 1, 1943, p. 32, or the *Iron Age*, March 25, 1943.

DISCUSSION

Written Discussion: By Leslie W. Ball, assistant technical director, Triplet & Barton, Inc., Burbank, Cal.

The radiographer's interest in stereoscopic methods is concerned with three factors, namely: methods, sensitivity and application. The author has provided valuable data on the first two of these factors. A minor point in the text is that in Fig. 2 there appears to be no point in stating the type of film or in plotting curves for different films.

In cases where the film is tilted instead of shifted horizontally relative to the tube, an added correction has to be made for the difference in image shift between defects located at the center and at the edge of the film.

In practice, information about the position of the defect relative to the surface of the part is required, for three reasons: first, to predict if the defect will show on machining; second, to determine if the defect is located in the highly stressed outer fibers of the part; and third, in the case of repair, to predict how deep the chipper will have to go.

In general, knowledge of the characteristics of castings or thick welds allows the radiographic interpreter to make fairly accurate guesses of the location of defects relative to the metal surface.

Oral Discussion

H. E. SEEMANN:² I wish to mention but one point in connection with Mr. Rigbey's interesting paper. It seems to me that the procedure described might better be called a "parallax" rather than a "stereoscopic" method. If the X-ray tube shift (or specimen shift) is used to provide two different locations of the point of interest in a radiograph, or in a pair of radiographs, so that measurements can be made and the position calculated, the word parallax is appropriate. A method in which the tube or specimen shift is used to provide the three-dimensional impression when the radiographs are viewed in a suitable holder is a stereoscopic method.

Author's Reply

Replying to Mr. Ball, actually there is no point in making different curves for different film unless very different cassettes are used and the distance plotted from the top of the cassette instead of the film as we did here. We use two types of film, Noscreen with 0.2-millimeter lead filter in a cardboard holder and High Speed film with two calcium tungstate screens. Consequently there will be quite a difference in the distance from the top of the cassette to the film in the two cases. The graph that we saw was for High Speed film. You noticed that the curves cut the vertical axis at about 5 millimeters below the origin and that is, I believe, quite an accurate measurement of that distance. If you lose your calipers by any chance, then you have a ready-made method, going about it the long way, of getting an accurate measurement of that quantity!

²Physicist, Kodak Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

In answer to Dr. Seemann, I see we are getting into trouble over terminology again. I had the same trouble last time when we tried to describe the double-exposure method. It was brought out in the discussion of that paper that the term "double-exposure" should be used in those cases where two exposures are superimposed on the same film. The term "stereo-radiography" should be reserved for separate exposures on separate films. I decided that the term stereo might be used here due to the fact that if the tube shift is correct, the only difference between this and medical stereoscopy, as the doctor calls it, is the use of the lead marker and the scratch on the film. Those two pictures can then be mounted on the stereoscopic viewing screen and observed in depth. It might be called a modification of the stereoscopic method which enables the flaw positions to be calculated by measurement as well as optically.

I would like to thank the discussers for their interest in the work described. The application of the method was not dealt with as that has been treated previously and the method, or modifications of it, used for many years. This work is, of course, avoided whenever it is possible to take a second radiograph at right angles to the first. It is a last-resort method to be used on parts of difficult shape.

The term "parallax" is a more general and consequently better term than "stereoscopic". As Dr. Seemann intimates, the latter applies only when the tube shift is of such dimensions that the films can be viewed in a stereoscopic stand without distortion. The correct perspective is obtained when the central rays of the two exposures converge at the same angle as that formed by your lines of vision at viewing distance. The greater the S.F.D. in relation to the viewing distance, the greater the stereo shift required. 2.5 times the S.F.D. divided by the viewing distance equals the correct stereo shift. A convenient table of tube shifts for stereoscopy appears below. It will be noticed that the small tube shifts required in the table would result in a greater percentage of error when making the measurements and hence a "parallax" method only would be generally employed.

Stereo or Tube Shift (Inches)				
S.F.D. Inches	Viewing Distance Inches			
	25	27	29	31
25	2½	2¼	2¼	2
28	2¾	2⅝	2⅝	2¼
30	3	2¾	2⅝	2⅜
36	3⅝	3⅝	3¼	2⅞
40	4	3¾	3½	3¼
48	4¾	4½	4⅝	3⅞
60	6	5½	5¼	4⅞
72	7¼	6¾	6¼	5¾

BASIC ELECTRIC MELTING PROCEDURE FOR HIGH QUALITY ALLOY STEELS

By A. L. ASCIK

Abstract

In this paper, the writer has described the melting problems of high quality alloy steels exclusively.

Reasons for the lack of agreement between the steel-makers as far as a standard melting procedure is concerned are discussed. Then, assuming that the heterogeneity of the ingot is not always clearly shown by photomicrographs and photomacrographs, and particularly that this property is not measured by figures, he takes as a determining basis of this heterogeneity, the difference between the longitudinal and transverse physical properties found in the forged ingots and points out the influence of the melting method on this heterogeneity. He describes the charge, melting and boiling periods, explaining the part played by the manganese-silicon ratio in the charge, the quantity of carbon and manganese remaining when the charge is melted down, the time and quantity of the ore addition, the boiling ratio and temperature, etc.

A definition of the white and carbide slags follows, their chemical and physical advantages and disadvantages being discussed while the decisive influence of the physical processes on the quality of steels is specially pointed out. The solution of nonmetallic particles in the fluid steel at a high temperature and the fact that they are not precipitated when the steel solidifies in the ingot molds is discussed.

Finally, he describes the addition of aluminum as harmful to the cleanliness of steel and a high silicon content as very helpful to the surface condition of the ingots.

REASONS FOR DIVERSITY IN MELTING PRACTICES

OBSERVATION of the technical literature, as well as of the different written and verbal discussions concerning electric melting procedures, discloses that many factors of decisive influence on heat or ingot quality have been interpreted in practice in many entirely different ways.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, A. L. Ascik, metallurgical engineer, is superintendent of Steel Plant, Sorel Industries, Ltd., Sorel, Canada. Manuscript received June 5, 1944.

There seem to be several factors preventing the achievement of complete unity of view among steel-makers in regard to the melting procedure.

One of these reasons is the fact that steel-makers, producing various grades of steels for different purposes, interpret the melting procedure in their own way, recommending some methods which are supposed to improve the steel quality. For example, the casting-makers or commercial quality steel producers who, in 95 per cent of the cases, are satisfied by remelting the scrap and "killing" the fluid steel as soon as elements are adjusted, discuss melting procedures with gun, aircraft or tool steel-makers, who, of course, could never obtain satisfactory results if they applied such procedures.

Such discussions darken the melting problems instead of enlightening them; they complicate the melting practice, particularly in the case of steel-makers making heats mechanically and by intuition rather than with a sufficient knowledge of what really occurs in the fluid bath during each individual period of the heat. They show an inclination to try any new suggestions but, applying them at the improper time and period of the heat, they are, in many cases, disappointed by the results obtained on trial heats. Consequently, the conviction that the "old method" was the best governs once more their melting practice.

Therefore, before entering into such discussions, a distinction should be made between the different ultimate uses of steels made in electric furnaces. Opinions could then be exchanged on the details of melting, supporting them by figures showing the metallurgical and economical results, as only on the basis of such figures can the best and most economical procedure for each particular case be found. This would also constitute a first step toward standardizing the melting procedure, which the writer strongly believes can be done and which would be most helpful to all steel-makers.

The second reason worthy of mention lies in the question: "Is steel-making an art or a science?" The opinion that steel-making is an art still exists in the minds of many people. However, close observation and careful experiments prove that it can be directed with almost theoretical accuracy from a scientific point of view. Particularly is this true in the case of the electric furnace, which is an apparatus with great chemical advantages, providing that the steel-maker is sufficiently familiar with them and applies them properly. The logic and hard eye of science directed on the hands and shovels

of the furnacemen, and at the same time checking the quality of the finished product, can eliminate many differences of opinion and, particularly, the "tricks" of steel-making.

The third and most important reason is the fact that in many cases, the steel plants themselves fail to define clearly the quality of steels. Most opinions concerning the final quality of steels come from the customers and this does not seem in order. The steel plants must have the ways and means to determine the quality of their steels on the basis of the melting procedure applied by their melting department; yet, steel-makers are found who do not even know the quality of the steels they are making. Recently, some of them with years of experience in the manufacture of quality steels met considerable difficulty when certain specifications required special tests as, for instance, that of the top and bottom of each ingot of the heat.

UNIFORMITY OF HEATS, THE MOST IMPORTANT AIM IN MELTING

Very important questions arise for any steel plant producing high quality steels: How to ascertain whether the applied melting procedure gives the best possible results, how to know which melting procedure is the best, and finally, what determines the quality of the heat?

It is well known in practice that individual heats, even though made in the same manner, do not show uniform results. One of the most important questions in steel-making, "What guarantees the uniformity of heats?", has not found its answer up to now. Undoubtedly, it depends upon the melting procedure applied and upon the accuracy with which all details of the melting procedure are performed. In practice, of course, errors are inevitable, particularly when melting disturbances occur and the furnace is out of the melter's proper control. All steel-makers would like to find a melting procedure which would assure a good heat and ingot, even though disturbances were met; or, at least, would diminish the influence of these disturbances on the quality of heats. They would also like to discover some kind of test which would verify any melting procedure before the bulk of the tonnage is melted; in other words, a test which, applied on trial heats made by different methods, would determine which melting procedure gives the highest percentage of good heats or the highest uniformity of heats.

The aim of this article is to outline the results obtained by a melting procedure applied by the present writer for several years to high quality alloy steels, suggesting at the same time a method to test the melting procedure as well as describing the melting procedure itself.

INGOT HETEROGENEITY A MEANS OF DETERMINING THE VALUE OF THE MELTING PRACTICE

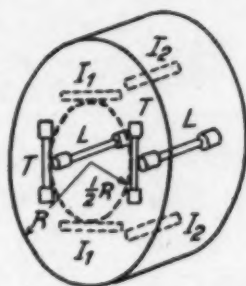
Melting is the first stage in which the quality of steel is created but, although some rules are applied during this procedure, there is, at the present time, no way of checking the quality of the heat during the melting period.

The next stage is that of the solidified ingot. The property which determines the quality of the ingot is its heterogeneity. Micro and macro tests, when applied and interpreted properly, prove to be most helpful in demonstrating the chemical and physical heterogeneity of the ingot; but nevertheless, they are not sufficient to find all the internal defects of steel. In many cases, it was found that steels made by various methods, in separate furnaces and showing different properties and qualities in the final products, appeared identical under micro and macro tests. Therefore, it would appear that although some defects are discovered by micro and macro tests, others, which seem to be of an organic and consequently submicroscopic nature, fail to be uncovered by them. Apparently, the nature of these defects depends upon the melting chemistry which is also the determining factor in obtaining a more or less heterogeneous ingot. All kinds of failures which the metallurgists often face without finding a clear reason for them, such as irregular fractures shown by the tensile test, low physicals, low impact figures, etc., are caused by the physical or chemical heterogeneity of the ingot. If a method were found to check the grade of this heterogeneity in a more definite manner, it would also be possible to verify the melting chemistry or the melting procedure.

DIFFERENCE BETWEEN LONGITUDINAL AND TRANSVERSE PROPERTIES, A MEASURE OF INGOT HETEROGENEITY

While looking for such a method, the following experimental investigation was made: a 25-inch square ingot was forged in steps

under a 2500-ton press, the ratio of reduction being 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20 to 1. The whole forging was cooled slowly during 5 days, being normalized and annealed afterward. It was cut in sections according to the forged steps and each section was heat treated individually to a yield point of about 45 tons. Tensile and impact tests in the longitudinal direction were then taken as shown in Fig. 1, and the difference between the longitudinal and transverse tests



L - Longitudinal Tensile Test
T - Transverse - - - - -
*I*₁ = - - - - - Izod's - - - - -
*I*₂ = Longitudinal - - - - -

Fig. 1—Diagram Showing Manner in Which Tensile Test Specimens Were Taken.

Table I

No. Heat	Refining Slag	Ladle Analysis of Heats—Per Cent								
		C	Mn	Si	P	S	Cr	Ni	Mo	Cu
1910	White	0.32	0.74	0.38	0.019	0.017	0.81	3.05	0.31	0.19
1916	White	0.34	0.78	0.41	0.021	0.018	0.79	3.11	0.31	0.16
1924	White	0.32	0.77	0.36	0.018	0.015	0.78	3.01	0.32	0.21
1929	Carbide	0.33	0.76	0.37	0.020	0.019	0.79	3.07	0.33	0.19
1934	Carbide	0.33	0.79	0.36	0.019	0.017	0.76	3.03	0.32	0.20
1938	Carbide	0.33	0.75	0.39	0.019	0.018	0.78	3.09	0.32	0.17
1941	Black	0.32	0.79	0.37	0.017	0.015	0.77	3.07	0.30	0.21
1946	Black	0.33	0.76	0.39	0.019	0.018	0.79	3.01	0.33	0.20
1950	Black	0.34	0.75	0.34	0.019	0.017	0.76	3.06	0.32	0.19

on each step was considered an indication of the ingot heterogeneity. The bigger the difference, the more heterogeneous the ingot and the worse the heat or melting procedure. Elongation, reduction of area and impact figures are particularly sensitive to this kind of testing.

These experiments were carried out on three different melting procedures made under the following refining slags: white, carbide and black. Three heats were made by each method. Boiling was applied in all cases. The tapping temperature was 1640 degrees

Cent. (2985 degrees Fahr.) and the pouring temperature 1580 degrees Cent. (2875 degrees Fahr.). The steel made was of grade S.A.E. 3330 with addition of 0.3 per cent molybdenum. Ladle analyses from these heats are given in Table I.

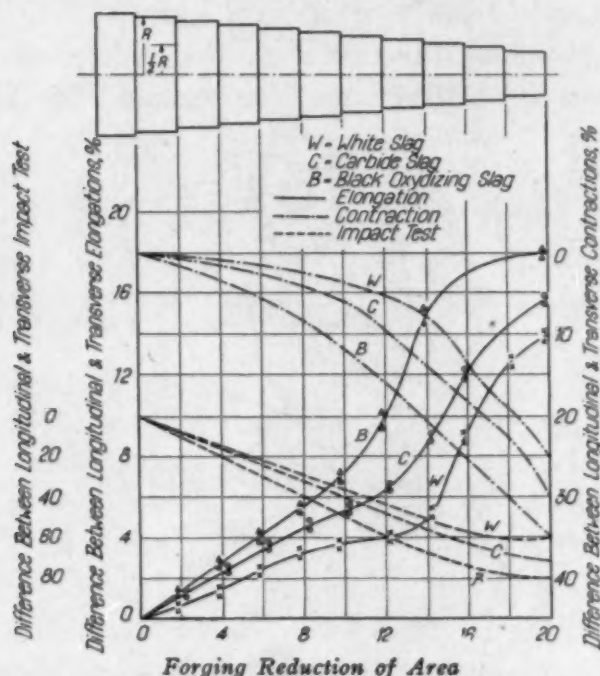


Fig. 2—The Difference in Longitudinal and Transverse Elongation, Contraction and Impact Tests Between Forged Ingots Refined Under White, Carbide and Black Slags.

Fig. 2 shows the final results on the basis of which a general conclusion can be made that the white slag method gives the highest and most uniform figures. Similar results were obtained by the writer in the course of his practice in several countries and plants. Whenever steels with high transverse physical properties or of the highest possible cleanliness were required, white refining slags gave the best results.

THE CHIEF FACTORS OF STEEL-MAKING

Before recounting further details of the procedure, let us point out certain factors concerning the melting procedure which are worthy of mention. They are more or less known but have not been sufficiently insisted upon either in the technical press or in daily practice.

First of all, it should be kept in mind that steel-making is a battle against oxygen, no matter in which state or shape this element

exists, in the scrap or in the fluid bath. This is practically the whole essence of the steel-making chemistry. The quality and quantity of oxidizers and deoxidizers, the time and manner of their addition, their mutual quantitative ratio, etc., all these factors belong to the "strategy" of the battle against oxygen, applied through the whole melting procedure, from the moment the first drop of fluid steel appears under the electrode to the time the first drop of tapped metal falls into the mold.

There are, in steel-making, both oxidation and deoxidation processes which are of entirely different chemical directions and which must occur simultaneously. The proper temperature at the proper period of the heat and during the proper length of time is the most effective factor in the hands of any steel-maker in his battle against oxygen.

Chemistry alone does not suffice to make a good clean steel. There are also some physical processes and, if they do not occur properly, the steel obtained is only of average or below average quality even through the chemical side of melting may be carried out in an almost flawless manner.

The following can be considered as physical processes: the degasification of the fluid bath which depends on the time and intensity of boiling; the rising of nonmetallic inclusions during both the boiling and the refining period; the fluidity of the slag and bath; the ratio of their adhesion; and the kind of surface contact between the slag and the bath. Upon these physical factors chiefly depends the effect of the refining period during which some deoxidation products are "thrown" out of the fluid bath and "caught" by the slag. The writer's own observation of the final results of heats has led him to believe that in the case of electric melting procedures, the physical processes often play a more important part than the chemical. The mutual relation of such little known properties of both the slag and fluid steel as fluidity, surface tension and adhesion, and their influence on the cleanliness of steels, seems to represent a particularly wide field of investigation.

The fourth factor is the necessity of keeping the melted steel as homogeneous as possible. Let us point out that this can be done only when the steel is in the furnace because in any subsequent stage of its manufacture, it is always internally segregated.

As a matter of fact, steel is not even homogeneous at the fluid stage—this can easily be seen when observing the boiling slag. When

a large quantity of iron ore is added, the slag foams and rises up due to the violent bath action. After 5 to 10 minutes, the level of the slag falls creating the impression that the boiling intensity has diminished because the iron ore is "boiled out". But, if instead of adding fresh ore a strong stirring is applied, the violent boiling action appears once more. This proves that the temporary disappearance of intensive boiling was not caused by the lack of iron ore but by the change of the carbon content on the surface or near the surface of the fluid bath. Therefore, the bath should be stirred, as boiling, which undoubtedly causes some mechanical movement of the fluid steel, is not sufficient to equalize the carbon content all over the bath. Moreover, practice demonstrates that when a fluid bath is kept without frequent and strong stirring, it gives almost invariably an ingot of only average quality.

Taking into consideration that the specific gravity of fluid steel is around 7.14 and that this kind of "fluid" is very dense, it can easily be seen that there could be in the bath "spots" of different temperature or of different chemical composition. Moreover, similar "spots" can also be created by the addition of alloy elements. The problem lies in the solution of these elements in such a way that they be evenly divided in the fluid bath. In order to obtain this even distribution, and to avoid a bath which is very undesirable, strong and frequent stirring should be applied.

Finally, the writer would like to underline a new factor which he believes can be called the "heat treatment" of the fluid bath. When the final results obtained on steel products are checked with the melting conditions, some indications are found which lead us to believe that some nonmetallic substances are dissolved in the fluid steel. The solution of these substances occurs slowly, yet once they are dissolved they are not precipitated again even during the ingot solidification. In other words, the ingot solidification can be considered as some kind of "quenching" of the fluid steel. This quenching seems to start at tapping temperature and to finish at solidus temperature. The advantage of this procedure appears in the higher quality of the macro test.

CONDITIONS OF A PROPER CHARGE AND MELTING PERIOD

Passing in turn to the details of the melting procedure, let us underline that it starts when the first pool of fluid metal appears on

the bottom of the furnace. If the scrap does not contain an excessive quantity of phosphorus, there is no need to take special precautions to remove it and the oxygen then attacks chiefly two elements: silicon and manganese. Oxides of these elements create a wide scale of silicates, from very fluid to very hard. Melting conditions would be ideal if these nonmetallics were removed from the fluid steel almost at the same time as the melting of the scrap progresses and the lake of fluid steel increases.

The rising process of silicates depends first of all on their fluidity and then on their size. The more fluid the silicates, the easier the process of their coagulation and rising. Investigations of Herty, confirmed by Körber, Olsner and others, show that in order to create fluid silicates, a certain ratio of manganese-silicon must be kept and that the lowest ratio is around 2:1. Applying this rule to the electric melting procedure, it is found that in the case of scrap containing 0.20 to 0.30 per cent silicon, a minimum of 0.50 to 0.60 per cent manganese is needed in order to create a fluid silicate. In reality, it is advisable to increase the theoretical ratio for the following reasons:

- a) To compensate for the quantity of manganese which evaporates while the charge melts (brown fumes).
- b) To assure the origin of fluid silicates.
- c) To protect the carbon against excessive oxidation as higher carbon losses during melting mean excessive iron oxidation which is deadly for any steel-making.

It is commonly known and found in practice by many steel-makers that heats which are melted with a low percentage of manganese are of poorer quality than those melted with a higher percentage. Low manganese heats mean that there are many small hard silicates which do not rise and do not coagulate and, being dispersed in the whole fluid bath, create some kind of suspension which changes entirely the nature of the steel. Its fluidity, rolling or forging properties, physical properties (particularly in the transverse direction) and many other properties are then quite different from those found when heats are melted with a higher percentage of manganese. Electrolytic analyses show that when steel inclusions contain over 40 per cent SiO_2 , all kinds of production difficulties occur and even the best boiling and refining operations do not help much to improve the heat.

What percentage of manganese should be obtained after the charge has been melted down? It seems that when the boiling period

is omitted, 0.30 per cent manganese in the first test should be considered a minimum. When there is a boiling period, the quantity of manganese should be higher as it drops during the ore addition. It would be false to believe that by saving on the quantity of manganese added to the charge, the cost of steel is diminished as practice shows that the expense of scrapping steel because of an insufficient quantity of manganese during the different stages of the heat is very much higher than the cost of the manganese.

One rule should therefore always be kept strongly in mind, that *the charge should contain an alloy in which the manganese to silicon ratio is on the side of fluid silicates.*

The second element which determines the quality of the heat is the carbon content remaining after the charge is melted down. While the common opinion is that a low carbon content, sometimes below 0.1 per cent, is preferable, the writer's opinion is that this practice, although successful for average commercial grade steels, does not give satisfactory results in the case of high quality steels.

Low carbon contents are obtained when iron ore is added to the charge or when the quantity of manganese in the charge is insufficient. In both cases two very undesirable reactions occur:

- 1) The strong oxidation of manganese when the presence of manganese is the indispensable condition for the fluid bath's self-cleaning during the melting period.
- 2) The more or less strong oxidation of iron when all oxidizing elements, including carbon, are low.

There is a general law for every steel-maker melting high quality steels: "Protect iron against excessive oxidation at any stage of melting". This is the essence of correct melting chemistry.

Once the iron is excessively oxidized, the refining period is so difficult that in many cases it is almost impossible to get a good heat. The reason why heats of low carbon alloy steels are considered the most difficult is that steel-makers are compelled to oxidize the iron excessively, as they must bring down the carbon content by adding more ore into the charge or boil down the carbon after the heat is melted. In both cases, it is unavoidable to introduce an excess of oxygen into the bath because the carbon oxidation process proceeds very stubbornly due to the fact that low carbon baths require higher carbon oxidation temperatures and more iron ore. If the bath is overheated and contains much dissolved iron oxide, the deoxida-

diately after it is melted. They believe that the melting and boiling procedures can occur at the same time. When the charge is melted down, they immediately take off the melting slag and start the refining period. This method is, of course, the most economical but it is hard to admit that proper and effective boiling was applied. The iron ore in the charge cannot cause proper boiling at the same time as the melting occurs. It has to oxidize silicon, phosphorus, manganese and after these elements have been lowered, it must react with the carbon. It is well known that the lower the carbon is, the higher the boiling temperature must be. At the same time, the bath, immediately after being melted, is not hot enough, so in order to cause boiling, it is necessary to increase the iron ore concentration in the charge and practice shows that all heats made by this method contain so much ore that the first test, taken after the charge is melted, shows very low carbon and manganese contents which, as said before, proves that the bath has been overoxidized or even "burned" while melted down. This is the main objection to the opinion that the boiling period can occur simultaneously with the melting period.

The principal factors upon which depends the success of the boiling procedure are the following:

- a) Time of boiling
- b) Temperature of boiling
- c) Quantity of iron ore used
- d) Rate of boiling.

Inasmuch as the removal of hydrogen is considered, various theoretical and practical investigations made during the last six or eight years show that the strong action of the bath during boiling removes practically all the hydrogen in 25 to 45 minutes. This time is also sufficient to change the state of the relatively cold dead melted bath to a strongly reworked homogeneous fluid. It is also sufficient to cause the coagulation of a certain amount of smaller silicates which did not rise during melting or to remove another amount of them by physical process. The latter reaction relies upon the intimate contact between the fluid steel and the slag when the boiling action occurs. The slag washes out an amount of nonmetallic particles which rise only with difficulty. As the temperature of the metal increases during boiling all these processes occur under more favorable conditions. In practice, the boiling period can last around 40 to 60 minutes while the boiling speed is around 0.18 to 0.25 per cent carbon boiled down per hour. This kind of boiling causes a

foaming slag which has a tendency to overflow through the furnace door.

The next condition for proper boiling is temperature. The essence of boiling relies upon the removal of carbon and this reaction occurs better under higher temperatures. In practice, the best boiling temperature exists when 1 to 1.25 points of manganese are oxidized for every point of carbon boiled down. In other words, when the rate of carbon oxidation is 0.20 per cent per hour, the quantity of manganese oxidized should be 0.20 to 0.25 per cent per hour. If the boiling temperature is lower, the manganese is oxidized rapidly and there is a danger that the oxygen will attack the iron. If the boiling temperature is too high, there appears another reaction which also gives the same results, i.e., iron oxidation, according to the following reactions:



When this kind of boiling occurs, the manganese oxidation is of a lower rate than that mentioned previously or else this element after reaching a certain minimum increases. As an example let us quote the following:

First test after charge was melted down.....	0.55 per cent manganese
Second test taken 30 minutes later.....	0.38 per cent manganese
Third test taken just before removal of slag....	0.43 per cent manganese

The final result of such boiling is the oxidation of the iron with the simultaneous letdown in boiling intensity due to the fact that the iron is attacked not only by the manganese oxide, but also by the added iron ore which, in reality, is destined exclusively to the removal of carbon and manganese. Although the problem of temperature conditions is one of the most important of the boiling procedure, it has been frequently neglected in practice. Experience shows that when boiling is carried out at the proper temperature, the refining period proceeds very smoothly and the heat always shows excellent properties in all respects.

Another point which should also be mentioned here is that when the boiling procedure occurs at a too high temperature it is entirely wasted as far as degasification is concerned. Heats which are boiled at a too high temperature, and which contain a too high quantity of manganese before the slag comes off, show the biggest tendency to being "wild" and the biggest sensitivity to "flaking". It would mean

that the removal of hydrogen is limited to a certain maximum temperature and once this temperature is passed, the fluid bath dissolves more hydrogen than the amount removed by boiling. It would also appear that this solution of hydrogen occurs very quickly so that sometimes 10 to 20 minutes are sufficient to have more hydrogen in the boiled bath than before boiling.

The quantity of iron ore to be added is strictly connected with the boiling temperature. In both cases where the boiling procedure is carried out at an either too high or too low temperature, an excess of iron ore has been added. The quantity of 4 to 6 pounds of iron ore for 1 pound of carbon gives the best results.

In summing up what was said above about the boiling period, the following conditions can be said to assure the best boiling procedure in the case of structural steels containing 0.25 to 0.40 per cent carbon:

- a) Boiling time 40 to 60 minutes
- b) Oxidizing rate of carbon..... 0.18 to 0.28 per cent per hour
- c) Oxidizing rate of manganese 0.18 to 0.27 per cent per hour
- d) Iron ore used..... 4 to 6 pounds per 1 pound of carbon removed
- e) Manganese content before removal of slag. 0.30 per cent minimum or 0.35 per cent maximum
- f) Carbon content before removal of slag..... 0.20 per cent minimum

If the boiling temperature will be such that all these conditions will be kept simultaneously, the difficulties in obtaining a good heat will be reduced to a minimum.

THE ADDITION OF IRON ORE AT THE PROPER MOMENT, AN ESSENTIAL FACTOR

Before bringing to an end the description of the melting and boiling periods, the writer would like to stress once more, this time from another point of view, how important it is that the iron ore be added at the proper moment. Fig. 3, representing the ternary diagram which depicts the viscosity relations, illustrates the following conclusions:

1. When ratio $\frac{\text{MnO}}{\text{SiO}_2}$ is higher than $\frac{68}{32}$ and lower than $\frac{78}{22}$ which corresponds to ratio $\frac{\text{Mn}}{\text{Si}}$ 4 and 7, fluid silicates can be created easily and the influence of the iron ore, even charged with

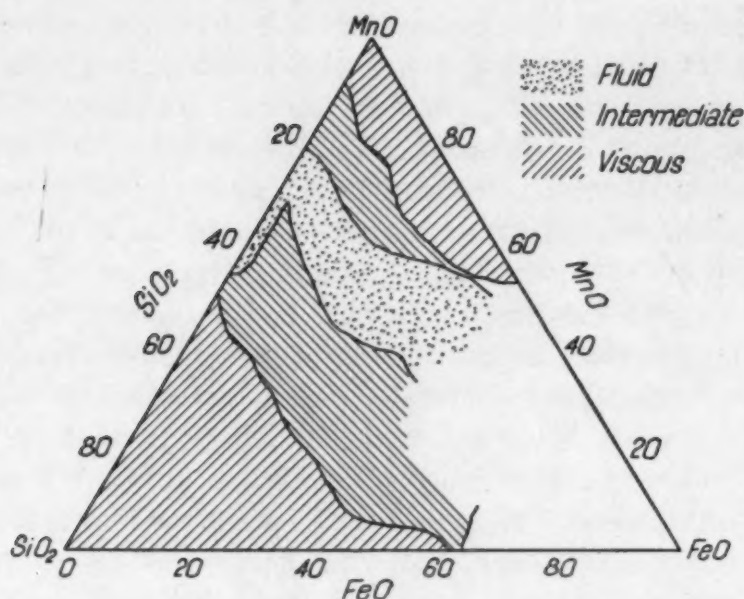


Fig. 3—Ternary Diagram Illustrating Viscosity Relations.

the scrap, is not to be feared. In practice, however, the scrap dealt with does not contain such a high manganese-silicon ratio.

2. When ratio $\frac{\text{MnO}}{\text{SiO}_2}$ is lower than $\frac{68}{32}$ and higher than $\frac{55}{45}$, which corresponds to ratio Mn 4 and 1.9 and which also represents the ratio used in practice, the quantity of iron oxides contained in the charge should not be over about 3 per cent as otherwise there is a possibility of creating intermediate silicates.

The fact that the scrap itself contains around this quantity of iron oxides proves once more that iron ore should not be added to the charge before the silicates are created or, in other words, before the charge is melted.

REFINING PERIOD

DIFFERENCE BETWEEN CARBIDE AND WHITE SLAGS

Before describing the details of the refining period, it might be useful to define "refining slags," as often some steel-makers use the definition "white" slag for "carbide" slag and vice versa. These two slags are really quite different in every respect. Carbide slag is a mixture of lime and spar which, after fusing, is deoxidized by carbon-containing substances such as coke, powdered electrodes, anthracite, etc. This slag, when cooled, is of a grayish color and it

emits a specific odor of acetylene when it is put into water. White slag is a mixture of lime, spar and sand which after fusing is deoxidized by 75 per cent powdered ferrosilicon. The color of this slag when disintegrated is white or very slightly grayish.

Theoretically, the best deoxidation occurs under carbide slag as its products are gaseous and are removed from the fluid bath immediately and permanently. Practically, however, this slag does not seem to give the best results as, under carbide slag, the percentage of doubtfully deoxidized heats is higher than under white slag. Apparently, this is due to the following reasons:

1. It is a well known fact in metallurgy that where chemical procedures occur between slags and fluid metals, the hottest slags are the most chemically active. Carbide slag, for two reasons, must be of an exceptionally high temperature: Its melting point is relatively high and reactions of deoxidation and desulphurization by calcium carbide occur better in higher temperatures.

Considering that the fluid bath and furnace are cooled during and after slagging, that the fusion of the mixture lime-spar takes a certain time and that, after it does become fluid it still remains cold for quite some time, it can be said that the chemical activity of the carbide slag during the first 25 to 35 minutes of the refining period is very low.

Moreover, as in commercial conditions, the refining period lasts from 1 hour to 1 hour, 20 minutes, the average 40 minutes remaining under fairly warm slag is not sufficient to get an effective refining.

In the case of white slag, conditions are quite different. A powerful exothermic reaction is obtained when the 75 per cent grade ferrosilicon is added to the slag. About 50 per cent of the addition is burned to SiO_2 , heating the slag to a temperature which can never be attained by a carbide slag. This fact, coupled with low fusing point, results in a very active slag within 10 minutes of the fine silicon addition. Consequently there is a corresponding increase in the refining action.

2. The physical homogeneity of the carbide slag is much lower than that of the white slag. It can be easily observed in the furnace when using both slags. This must undoubtedly have an influence on the chemical activity of the slag which, when heterogeneous, does not react uniformly with the whole surface of the fluid bath.

3. The deoxidation by carbide slag is due exclusively to the contact of the slag with the bath. The more intimate this contact

the better and faster the deoxidation reactions. As deoxidation products are gaseous, they have a natural tendency to rise and when doing this, they move the slag over the bath; at this moment, the contact slag-bath is ruptured. This process is continuous and occurs in numerous places causing a foamy slag. These conditions are not favorable to reactions which first of all depend upon the contact of two substances. This disadvantage does not exist in the case of white slag. The bath is deoxidized by ferrosilicon which goes through the slag into the bath, keeping the contact slag-bath very intimate.

4. Carbide slag, by nature, is not a very strong deoxidizer. When it becomes carbide, it does not mean that the bath has been sufficiently deoxidized but only that some kind of equilibrium has been established between the slag and the bath. In other words, the slag contains a certain quantity of iron oxide which is in chemical equilibrium with a certain quantity of oxygen in the fluid steel. These quantities, of course, depend upon the temperature. Carbide slag under the best deoxidation temperature still contains about 0.4 per cent FeO. In the case of white slag, however, this quantity can easily be 0.1 per cent which corresponds to a lower oxygen content in the bath. This means that in the case of carbide slag, we approach the moment of final deoxidation (addition of ferrosilicon to the bath before tap) with a much higher oxygen content in the bath than in the case of white slag where some silicon is introduced in the early stages of the refining period. This is the essential difference between the refining processes under white and carbide slags.

When the final silicon addition is made shortly before tapping, the fluid steel is of almost tapping temperature. According to Chipman's diagram, Fig. 4, the higher the temperature at the moment of deoxidation, the greater the free oxygen equilibrium in the bath. In the case of white slag, the deoxidation by silicon is started immediately after the refining slag is melted. At this stage, the temperature of the fluid bath is the lowest and, consequently, the quantity of free oxygen in equilibrium must also be the lowest.

An objection, from the theoretical point of view, can arise here against the statement that steel refined under white slag contains less free oxygen than steel refined under carbide slag when in both cases, shortly before tapping, there is the same silicon content and the same bath temperature. Practice, however, shows that steel made under carbide slag contains, in the above mentioned conditions, 50 to 100 per cent more oxygen than steels made under white slag. This is

due to the fact that the concentration of the equilibrium ingredients does not change immediately after the temperature is changed. Fluid steel has a very high degree of inertia so that, once the fluid steel is deoxidized at a low temperature, it maintains less free oxygen, even if heated up to a higher temperature afterwards.

The writer's own investigations show that steels refined under white slag contain around 0.004 to 0.006 per cent oxygen and steels

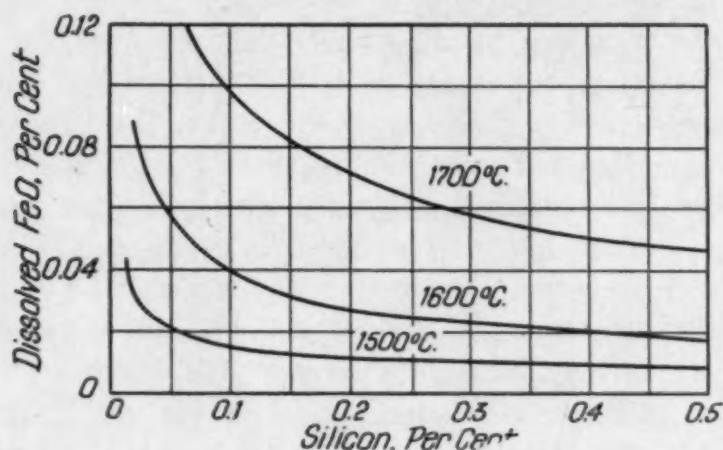


Fig. 4—Silicon-Oxygen Equilibrium Diagram (Chipman).

refined under carbide slag 0.009 to 0.012 per cent oxygen. These figures were recently confirmed by the Carnegie Institute of Technology which found that steel made under white slag, at the writer's present plant, contained 0.004 to 0.005 per cent oxygen.

5. The action of carbide slag in creating some kind of foam lowers its viscosity. Consequently, such an important physical process as the scouring out from the fluid bath of nonmetallic particles is not so intensive as in the case of white slag which adheres closely to the bath.

6. When examining under low magnifications the fractures of heat treated disks prepared from structural steels made under carbide slag, numerous small, white, shining cavities can very often be observed. There exists a thesis that these are caused by precipitated carbon monoxide dissolved in the fluid steel during the refining period. Some steel-makers, when making steels under carbide slag, always keep the furnace door slightly opened in order to avoid strong carbide slag and excessive pressure inside the furnace as both these factors favor the solution of carbon monoxide. Of course by so avoiding excessive pressure and strong carbide slag, they diminish the

chemical action of the slag as cold air introduced into the furnace cools the slag surface. A more effective method would be to have larger peep holes in furnace doors, and keep them constantly clean. The possibility of a carbon monoxide solution in the fluid steel and its further precipitation should also be regarded as a disadvantage of the carbide slag.

It may be concluded from the foregoing in regard to the refining period, that white refining slag is superior to carbide refining slag for the following reasons:

- a) The deoxidation procedure starts when the bath is at the lowest temperature.
- b) The contact slag-bath is more intimate.
- c) The slag contains less iron oxide.
- d) The bath is killed long before tapping thus allowing more time for the physical process of removing the deoxidation products.
- e) The slag attains a higher temperature which is the cause of its higher reacting effectiveness.
- f) Silicon is introduced into the bath gradually so the ratio of manganese-silicon is favorable to the formation of very fluid and easily coagulating silicates.
- g) The high viscosity of the white slag facilitates the physical processes of washing out the nonmetallic particles.

HEAT TREATMENT OF THE FLUID BATH

When very high physical properties are required, particularly in the transverse direction, steel, which after different metallurgical investigations does not seem to present any objectionable features, sometimes shows more or less irregular physical properties and fractures. Special attention paid to the pouring practice, the shape of the mold and ingot as well as to the heat treatment of the rolled or forged products does not solve these problems and, in most cases, the statement, that intercrystalline or interdendritic fractures cause the failures, closes the question.

Undoubtedly, the definition "intercrystalline" or "interdendritic" fracture is right and this kind of fracture is caused by some organic defects in the steel. Long investigations during which the melting department and laboratory worked in close co-operation were carried on in an endeavor to find the reason for these defects. As a

result, we dare to say that the fluid steel before it is tapped must be submitted to some kind of a heat treatment in the melting furnace. As in any heat treatment, there are some critical temperatures above which a sphere of solution exists and there are also one or more ingredients which, once dissolved, are not precipitated during all further processes of the steel manufacture. If the process of cleaning the fluid steel by creating fluid silicates and that of their raising and washing out by slag are considered more closely, it will probably be correct to say that all silicates are not made fluid and all fluid silicates do not rise. Fluid steel contains in suspension numerous submicroscopic nonmetallic particles of certain types, which never rise. In order to visualize better this phenomenon, let us say that this suspension is created as though the steel were turbid when fluid.

The question now arises of how persistent this suspension is when the temperature of the fluid steel is raised. It appears that these particles are dissolved in fluid steel and that the quantity dissolved depends upon the temperature. Besides the suspension created by silica or silicates, the small quantities of phosphorus and sulphur in the steel also constitute a problem which, as yet, has not been very well explained.

Nobody pays special attention to these elements considering that their average content in steel is about 0.02 to 0.025 per cent. However, it seems that this problem cannot be overlooked. It is well known that both these elements create phosphorus-sulphur-oxygen eutectics. When they are present in larger quantities as for instance in pig iron, they are usually precipitated on the grain boundaries.

In the case of steels, however, we can meet them in both shapes: dissolved or precipitated. This depends upon the temperature existing during the melting procedure. It is a well known fact that when making steels of high machinability, containing high phosphorus and sulphur, both these elements should be added at a high temperature. When added at low temperatures, their combination with oxygen creates a distinct network on the grain boundaries. Ingots of such structure very often crack when rolled. The fact that the network may be more or less indistinct or, in the best of cases, not exist at all, proves that these elements can be dissolved in steel with no precipitation following. The solution process depends only upon the temperature.

Transferring these observations to steels containing normal quantities of phosphorus and sulphur, we can suppose that there is

too little of each of these elements to create large coagulated precipitations placed on grain boundaries and that small quantities existing are precipitated and dispersed rather on interdendritic boundaries. This weakens the cohesion between dendrites and, as a result, gives irregular fractures and lower physical properties. Some experiments concerning the influence of the refining and tapping temperatures on fractures, physical properties and macro tests were made in an effort to avoid the sulphur-phosphorus chemical combination being precipitated or dispersed on the boundaries of the dendrites and keeping also in mind the soluble suspension of submicroscopic silica and silicate particles. It was found that the higher the tapping temperature, the better the macro tests, the more regular the fractures and the higher the physical properties (particularly in the transverse direction). Moreover, it was found that fluid steel must be kept at a high temperature at least 30 minutes before tapping. Finally, the degree of temperature depends upon the grade of steel and chiefly upon the carbon content which, as is known, decides the temperature point on the solidus curve. This is very similar to quenching temperatures which lie above the A_{c_3} .

Inasmuch as the grades of structural steels are considered, it seems that there are two critical temperature points: one around 1605 to 1610 degrees Cent. (2920 to 2930 degrees Fahr.); another around 1630 to 1635 degrees Cent. (2966 to 2975 degrees Fahr.). The structural steels tapped at 1610 degrees Cent. (2930 degrees Fahr.) or below generally show poor macrostructures; tapped at 1635 degrees Cent. (2975 degrees Fahr.) or below, they show fair macrostructures, and tapped above 1635 degrees Cent. (2975 degrees Fahr.) they show excellent macrostructures. At the same time, physical properties and fractures are considerably improved. Fig. 5, a, b, c, presents three macro tests of three different heats tapped: (a) at 1610 degrees Cent. (2930 degrees Fahr.); (b) at 1635 degrees Cent. (2975 degrees Fahr.); (c) at 1650 degrees Cent. (3000 degrees Fahr.). The grades of steel poured are those shown in Table I. All three heats had the same charge consisting of rolled crop ends from the same heat made in an open-hearth furnace, and were practically made in the same way until the refining period. The heat represented by Fig. 5 (a) was refined under white slag at 1610 degrees Cent. (2930 degrees Fahr.) during 1 hour, 30 minutes; the heat represented by Fig. 5 (b) was refined under similar slag but the temperature 35 minutes before tapping was 1630 degrees Cent.

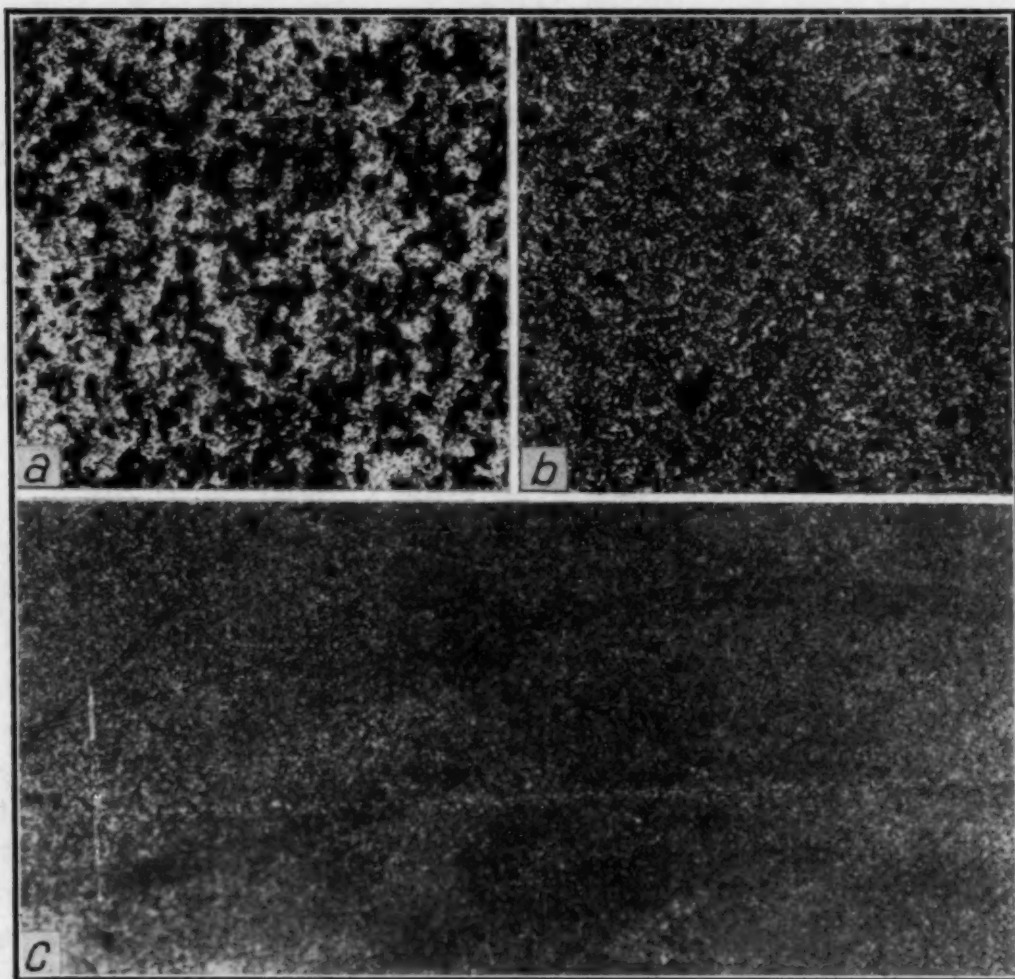


Fig. 5—(a) Macro Test of the Ingot Tapped at 1610 Degrees Cent. (2930 Degrees Fahr.). (b) Macro Test of the Ingot Tapped at 1635 Degrees Cent. (2975 Degrees Fahr.). (c) Macro Test of the Ingot Tapped at 1650 Degrees Cent. (3000 Degrees Fahr.). All pictures were taken from top of the 25-inch ingot forged for 12-inch round and on 2-inch radius from the center of the ingot.

(2966 degrees Fahr.) when measured on the spoon and 1635 degrees Cent. (2975 degrees Fahr.) in stream; the heat represented by Fig. 5 (c) was heated up to 1650 degrees Cent. (3000 degrees Fahr.). We concluded from this experiment that high temperature at the end of the refining period causes the solution of a considerable quantity of nonmetallic particles of larger or smaller concentration, which are usually discovered by macro test and that once they are dissolved, they are, practically speaking, not precipitated even during the ingot solidification. Of course, the bigger the ingot and the longer the solidification period, the more probable the precipitation of these particles. Practice shows, however, that the precipitation is practically insignificant up to about a 32-inch ingot.

This process of heat treating the fluid steel is, in certain cases, strictly connected with the results of the normal heat treatment. We find that when the heat is properly treated in the melting furnace, the soaking time before the normal quenching can be cut by 20 to 30 per cent. Also, the tempering times are considerably cut and the products show very uniform physical results.

INFLUENCE OF STRONG DEOXIDIZERS, PARTICULARLY ALUMINUM, ON THE CLEANLINESS OF STEEL

When melting high quality steels, the deoxidation method is the principal determining factor of the cleanliness of steel. Based upon many observations, the writer is of the opinion that the manganese-silicon deoxidation method gives the cleanest steels. Both these elements are natural and basic constituents of all steels and when intelligently handled, they give the cleanest steel. As mentioned before (Chipman's diagram), these two elements alone do not completely deoxidize because of a certain quantity of residual oxygen remaining in the fluid steel. This dissolved oxygen, however, has not much influence on the physical properties and even less on the cleanliness of the steel. It is probably precipitated during the ingot solidification and reacts with the carbon, silicon, manganese and iron but the products of these reactions being very finely dispersed in the whole mass of steel do not create any larger coagulation as the steel in the ingot mold freezes relatively fast.

A quite different phenomenon appears, however, when, by the addition of aluminum, a stronger deoxidation occurs. As the chemical affinity of this element to oxygen is several times higher than that of silicon, it reacts with the residual oxygen creating solid aluminum oxides which do not rise. They remain in the steel permanently and, which is worse, increase in size by creating aluminum silicates.

This reaction is particularly dangerous when the silicon is added just before tapping. At this moment, the steel is at the highest temperature and according to Chipman's diagram, the quantity of residual oxygen after the addition of silicon is very high. The aluminum addition which follows reacts with the oxygen and all efforts of the steel-maker to obtain a clean steel are in vain. An even worse practice is sometimes applied when the silicon and aluminum are added together in the ladle. When this kind of final killing is

applied, it is almost certain that transverse physical properties and magnaflux tests will show less than average results. Similar results are met when using certain ferro-alloys, as some of them contain as much as 16 per cent aluminum. The writer has had an unpleasant experience in manufacturing aircraft steels and forgings (exhaust valves) when using certain grades of ferrovanadium or ferrotitanium containing high quantities of aluminum.

Nevertheless, it seems that aluminum can be used with success if two conditions are carefully watched: that the bath already containing some silicon be as cold as possible and that the aluminum be added as long as possible before tapping. In practice, the procedure would be as follows: after slagging, about 0.15 per cent silicon would be added to the bath containing the aimed quantity of manganese and 10 to 15 minutes later, the necessary quantity of aluminum. However, this quantity should not exceed 0.04 per cent. The writer cannot recommend this procedure as a certain one as he has never applied it on a large tonnage of melted steel, but the addition of aluminum must be carried out carefully.

HIGHER SILICON CONTENT IN FINAL ANALYSIS, A HELPFUL FACTOR

Finally, a word should be said about the quantity of silicon in steel. Almost all specifications forecast the silicon content as 0.30 per cent max., some of them 0.35 per cent max. Keeping these limits in mind, the steel-makers add quantities of silicon which result in the final analysis showing a silicon content below 0.30 per cent.

Yet, nobody can explain or prove whether or not, in the case of structural steels (those for carburization excepted), a silicon content as high as 0.45 per cent is harmful. On the contrary, from the steel-making point of view, this element shows great advantages. Close observations prove that steels containing over 0.33 per cent silicon show the following advantages:

1. A spoon test poured in a small mold shows a very smooth steel solidification without any turmoil.
2. The ingot surface is better.
3. There are many less blowholes under the ingot surface.
4. The fluid steel is not so greatly affected by poor mold preparation.

It would, therefore, seem that steel-makers should require a change of specification as far as silicon is concerned and 0.45 per

cent as a maximum would not introduce any complications either in the heat treatment or in any other physical properties of the steel but on the contrary, would be of great help to steel-makers who experience great difficulties in ingot conditioning in the manufacture of high quality steels.

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DISCUSSION

Written Discussion: By T. W. Hardy, vice-president and director of metallurgy, Atlas Steels, Limited, Welland, Canada.

We would like to congratulate Mr. Ascik on the presentation of an interesting paper on a very important subject. As Mr. Ascik was formerly a valued member of our organization and since considerable tonnage of gun steel was made in our plant according to his ideas and under his personal supervision a brief discussion of the practical results obtained may be of interest at this time.

Since a perusal of Mr. Ascik's paper indicates that he has modified his ideas and practice since he left us, it should be emphasized that these remarks refer to the practice followed by Mr. Ascik at our plant about two years ago. The salient feature of that practice may be summarized as follows:

Melting and Oxidizing Period

(1) When melted, the bath should contain 30 points more carbon than desired in the finished steel.

(2) A high content of manganese was maintained throughout the boiling period, which was carried out at high temperature.

(3) Approximately 30 points of carbon were boiled out over a period of about 1 hour, ore being added in small lots.

(4) After about 20 points of carbon had been boiled out, about $\frac{3}{4}$ of the slag was removed. Fresh lime, spar, and sand were added and when the carbon had been reduced to the desired extent a complete "slag-off" was made, care being taken not to reduce the carbon any lower than necessary. At the time of slag-off the carbon content of the bath was about 5 points under the range (30-35) and the manganese content about 5 to 10 points higher than the carbon.

Refining and Finishing Period

(1) After the slag-off, a new slag was made of lime, sand, and spar.

(2) Manganese to the low side of this range was added as soon as a fluid slag was obtained.

(3) Finely ground ferrosilicon (75 per cent grade) was added to the slag to deoxidize it. No carbon in any form was used for this purpose. A feature of Mr. Ascik's practice was that as soon as this slag began to shape up, silicon from the slag found its way into the steel, which became progressively deoxidized at an early stage in the refining period. Consequently, additions of silicon at the end of the heat were not ordinarily made.

(4) Heats were held under a white slag for a minimum of 2 hours at a temperature that increased gradually from 2900 degrees Fahr. (1595 degrees Cent.) at slag-off to 3020 degrees Fahr. (1660 degrees Cent.) at tap.

(5) Mr. Ascik was opposed to the use of aluminum as a final deoxidizer.

It is a matter of record that a large number of heats were made according to this practice under Mr. Ascik's supervision in our plant and that these were generally characterized by exceptionally fine transverse properties. On the other hand, it is also a matter of record that during the spring and summer months when the humidity was high we had an abnormally high percentage of heats that had to be scrapped due to "hydrogen wildness".

It has been demonstrated beyond doubt in our plant, that Mr. Ascik's method, as then practiced, gave us steel that was much more susceptible to gassiness than that produced by methods more generally used on this continent. This fact, plus the practical objections that the long times and high temperatures involved were excessively hard on the furnace refractories, resulted in our abandoning the method in favor of our present more or less conventional practice which is giving us good physicals without any difficulties due to gas.

In conclusion we should like to point out that there is not the wide difference between Mr. Ascik's white slag and the so-called carbide slag that Mr. Ascik's paper would seem to suggest. Many operators use fine silicon with or without carbon to shape up their finishing slags, especially on low carbon steels, and the use of a slag containing appreciable amounts of carbide is by no means general on this side. The use of fine silicon in this manner greatly speeds up the deoxidation of the slag, but ordinarily it is not used in sufficient amounts to permit any appreciable increase in the silicon content of the bath. The important feature of Mr. Ascik's finishing practice on the other hand is the progressive deoxidation of the bath by fine silicon introduced slowly through the slag, resulting in the early incorporation of an appreciable silicon content in the bath and a problem in silicon control.

As was mentioned at the outset of this discussion a perusal of Mr. Ascik's paper indicates that he has modified his ideas and no longer insists on boiling in every case, and that he no longer advocates a partial slag-off during the boil. It also appears that the temperatures recommended for boiling and refining have been lowered and the time of refining shortened. It would be interesting to know if these changes were prompted by a desire to eliminate or moderate the gas trouble.

Written Discussion: By Gilbert Soler, superintendent of quality, Steel and Tube Division, The Timken Roller Bearing Company, Canton, Ohio.

The author is to be commended for presenting an outstanding paper on

electric steel-making. His paper emphasizes the extreme importance of attention to the many details required in successful melting practice. Certain of the author's scientific explanations of his practical observations are not in agreement with commonly accepted theories of physical chemistry. However, in the main, Mr. Ascik has proven his philosophy, that the art of steel-making can be largely reduced to a science by close observation and careful experiments.

The ladle analyses of the nine experimental heats shown in Table I indicate the ability to duplicate chemistry to a narrow range. It is surprising that phosphorus and sulphur contents should be so nearly alike for heats melted under white, carbide, and black slags, respectively. If analyses of tapping slags are available, it is suggested that they be included in the data tables.

Examination of the physical properties illustrated in Fig. 2 shows that forging reductions in the range 8:1 to 14:1 increases the difference between longitudinal and transverse properties of steels made under black slags at a greater rate than steels made under white slags. Similarly, the same properties are obtained with white slags at 12:1 forging reduction as are obtained from black slags at 6:1 forging reduction. Does this indicate that mold size and reduction in section by forging has a greater influence on physical properties than melting practice?

The value of stirring the bath frequently in producing homogeneous metal is well emphasized by the author. Some physical chemists may differ in their explanation of the mechanism, feeling that stirring provides a surface to initiate the carbon boil rather than attributing the action to differences in carbon concentration in the bath. Some physical chemists may also disagree with the author's statement that certain nonmetallic oxides such as silicates can be dissolved in the steel and prevented from later precipitation during solidification by the quenching action of the mold. There is, however, no question that a heat of steel should be tapped hot in order to obtain clean steel as judged by the macro etch test.

Mr. Ascik discusses thoroughly the differences between carbide and white slags and their relative deoxidizing action. He states that steel made under carbide slag contains 50 to 100 per cent more oxygen than found under white slags. Is this conclusion based on oxygen analyses taken from rapidly-cooled samples of molten metal or from slow-cooled samples? Steel-makers in the United States, finishing heats under carbide slags, in many cases combine this slag practice with early deoxidation at slag-off using an addition of 0.10 per cent silicon or 0.025 per cent aluminum. This practice will insure a low initial oxygen content under the finishing slag. As to sulphur and phosphorus, steel-makers will agree that these elements should be kept low and grain boundary precipitation should be avoided.

Aluminum additions are required for most steels produced in the United States in order to meet grain size specifications. Additions are made to the furnace if at all possible. By careful attention to the timing and order of aluminum additions, clean steel has been made to close magnaflux specifications in considerable quantity by the major producers of aircraft steel.

Most specifications for silicon content of alloy steels require 0.35 max. It is agreed that high final silicon is helpful under certain conditions; especially

when aluminum is not used. High silicon content of 0.45 silicon or more may result in a tendency towards brittleness in certain medium carbon high alloy steels. This is indicated in certain of the NE 9000 series steels.

Oral Discussion

W. J. REAGAN:¹ This paper is interesting and makes an interesting picture of alloy steel production. However, I do not believe that the American electric steel-making fraternity can accept many of these data as a procedure to make quality steel. Many thousands of tons of steel of similar types have been made in this country with an entirely different practice and their steel has met all quality specifications demanded. When we attempt to tie down certain factors to melting practice we find so many variables that it is impossible to find any correlation.

I am sorry that there are not a great many figures for many heats showing slag analyses, metal analyses and a log of a number of heats. To this should be added physicals on many heats and then we can form some conclusions.

As far as transverse properties are concerned we find a very small tonnage of electric furnace steels made in this country which are subject to transverse physical tests.

Regarding the manganese-silicon ratio in the charge, I can see no value from this item. In the first place in a tonnage shop with the wide analyses of scrap now being used it is impossible to maintain this ratio. Secondly I do not believe the author has presented data to prove this ratio of value.

Regarding high manganese as being an indication of clean steels, we often find that high manganese indicates a lack of sufficient oxidation in that heat with an indication of dirty steel.

Regarding the writer's comments on a vigorous boil, we believe in it 100 per cent, knowing it is one of the essentials to clean steel.

Confirming Mr. Soler's comments, there have been many thousands of tons of steel made with aluminum additions to control grain size, and quality has been all that was desired.

I can see no need of higher silicons in our steels as high silicon produces brittleness and furthermore when a large percentage of our steels have substantial additions of aluminum we have little need for higher silicons.

Regarding the oxygen in our steels, we learned from a paper given by John Marsh on yesterday's program² that oxygen in the steel, both under the oxidizing and finishing slag, is a function of the carbon in the bath, all of these figures for oxygen of course being determined before any deoxidizing materials have been added to the bath.

Furthermore, we still have no idea whether a 0.005 per cent oxygen or a 0.01 per cent oxygen is preferable in making quality steels.

I believe the author has made a lot of assumptions that have no data to back them.

I would like to ask the author what the tons per hour output of these furnaces is in making the type of steel discussed. A great many plants in this

¹Research metallurgist, Copperweld Steel Company, Warren, Ohio.

²J. S. Marsh, "Slag-Metal-Oxygen Relationships in the Basic Open-Hearth and Electric Processes," American Institute of Mining and Metallurgical Engineers Technical Program, National Metal Congress, Cleveland, October 18, 1944.

country have made similar steels at rates of 10 to 12 tons per hour and the writer has had experience in making similar types by a special process at rates of 20 to 25 tons per hour.

Author's Reply

Mr. Hardy points out that while we worked together, we used a longer melting procedure than the one mentioned in this paper. As he will remember, we experienced considerable difficulty in meeting the specified physicals when we started to produce the forgings from which the transverse tests were taken—40 to 60 per cent of the quantity tested were rejected on the first test. This restricted the capacity of the heat treatment department, cutting it practically in two, and increased the testing costs. At that time, the writer suggested a melting procedure which was sure to give better results. It was longer than the normal procedure, but was advisable under the circumstances, in order to produce as many forgings as possible which would pass the tests. The records show that under this procedure, over 95 per cent of the forgings passed the first test. Considering the specifications to be met (two tensiles, two impact tests and two bend tests from each end of ingot), this result was considered a big improvement on the former situation. We made about 20,000 tons of steel without losing one ingot because of physical properties or wildness.

The writer was indeed surprised that, during early summer, we had some wild heats due to hydrogen wildness. Unfortunately, he was unable to pursue his investigations along these lines, as he was transferred to another plant by Government authorities.

After having observed the steel-making conditions on this continent, the writer agrees with Mr. Hardy that the humidity of the air has a very big influence on the final quality of steels, particularly when plants are located near lakes or big rivers. Observations show that more reducing conditions kept in the furnace result in more hydrogen dissolved in the steel. These reducing conditions, of course, occur not only in the case of white slag, but also in the case of carbide slag. The writer believes that any steel plant which has to produce high quality steels should observe closely all atmospheric conditions.

The writer does not agree with Mr. Hardy that there is not a wide difference between white slag and so-called carbide slag. The deoxidizing procedure is quite different in each case. When white slag is used, silicon acts on the bath, giving as a result SiO_2 which decreases the basicity of the slag and increases considerably the temperature of both the bath and slag due to highly exothermic reactions. This causes both the bath and slag to become very active as far as chemistry of steel-making is concerned and their intimate contact created by these conditions cleans the bath very effectively.

Carbide slag represents a method of slow deoxidation, the products of which are gaseous. One of the purposes of the writer in presenting this paper was to investigate, by the preparatory work, whether or not various deoxidation methods known in practice give any significant differences in the ingot quality. It seems to the writer that the results presented suggest quite strongly that the three deoxidation methods used give very different results.

Some operators use fine silicon especially, as Mr. Hardy says, on low carbon steels. They usually do this in one of the following instances:

1. When the bath is overoxidized when melted or boiled and it is very difficult to obtain a disintegrating slag at the standard time of the procedure.

2. In the case of low carbon steels, fine ferrosilicon is used rather as a matter of precaution than for necessity as the operators fear that the excess use of coke or carbon will carburize the bath of low carbon contents and make the heat off analysis.

As Mr. Hardy will remember, the writer never insisted on a boiling procedure in every case, and it is a matter of record that while working together, we made a bulk of steel tonnage without boiling and we obtained very good results on transverse physicals. Following this, the boiling period was dropped in the case of smaller ingots.

The modified procedures presented here were applied to the conditions of this continent. Using one of them for a long time, the writer had about 0.8 per cent of scrap due to hydrogen wildness. This, however, was greatly compensated for by better physicals and lack of many reheat treatments.

Replying to Mr. Soler's discussion, the writer dared to explain, in a scientific way, his practical observations as he did not intend to ignore some very interesting phenomena of steel-making and, moreover, considering that all steel-makers feel that many very essential problems and factors in steel-making have yet to be clearly explained. The writer's wish was to offer an hypothesis to this audience for the purpose of hearing its reactions.

Scrap with low phosphorus and sulphur contents was especially used for these nine heats in order to avoid both dephosphorizing and desulphurizing procedures as the main purpose of the investigation was to find the influence of the deoxidation methods on the internal ingot structure. The scrap consisted of rolled carbon steel crop ends.

Analyses of the tapping slags were made only for typical elements and gave the following results:

	SiO ₂ Per Cent	FeO Per Cent
White slag	28 to 32	0.1 to 0.14
Carbide slag	20 to 24	0.40 to 0.45
Black slag	19 to 22	4.0 to 6.0

The difference between longitudinal and transverse physicals (when the same forging reduction is calculated) is higher in the case of dirtier steel. When speaking of dirty steel, the writer does not refer to certain nonmetallic inclusions visible under the microscope. This is perhaps one of the factors which should be the least considered as it appears only sporadically in steel plants of longer steel-making traditions. The matter is that the reason for dirty steel lies, in most cases, in the whole mass of steel or in the whole solidified ingot.

We mention in our paper that steel-making is a battle against oxygen and we especially used three slags containing different oxides in order to find its influence on the homogeneity of ingots. It seems that the part of oxygen is not finished when the steel is tapped into the ladle but that it also plays a large part when precipitating during the solidification of the ingot, although

it has in its vicinity (in the steel) some deoxidizers which are supposed to kill any of its tendencies to life.

The better or worse macro etchings of the ingot, its physical and chemical segregations, some kinds of internal laminations like ferritic stripes, etc., seem to depend on the oxygen content in the tapped steel and the form of its precipitation when the ingot solidifies.

With regard to forgings tested in the transverse direction, there exist, in steel plants of older forging traditions, some rules concerning these physicals. All of them agree that the higher the reduction of area the greater the difference between longitudinal and transverse physicals. Some of them, however, and even in the case of ingots of similar size, say that the forging reduction of area 4 to 1 is critical and that when this range has been crossed, the transverse physicals drop greatly. Some plants do not fear to use the range 6 to 1 and even 10 to 1 without any notable loss on physical values, when compared with the range 4 to 1. The writer's opinion is that these rules depend only on the cleanliness of the steel ingots made by these plants and that there are some differences in the melting methods applied by each plant, which widen or narrow the forging reduction of area but still maintain good physicals. The writer used the same mold sizes in his investigations, but different deoxidation methods, and found that the melting practice has a definite influence on the differences between transverse and longitudinal physicals, as the melting practice gives a cleaner or dirtier ingot.

It is quite possible that some physical chemists disagree with the writer's statement that certain nonmetallic oxides can be dissolved in the steel, and prevented from later precipitation during solidification, by the quenching action of the mold. There is, however, a law in physical chemistry that the solubility of a certain ingredient depends not only upon its known natural solubility, but also upon the size of the ingredient. In addition to this, if we consider the temperatures under which steel-making procedures occur, and if we also point out the fact that, until now, we knew little or nothing of the chemical or physical procedures at high temperatures, but at the same time, that there is a definite improvement in macro tests taken from ingots poured at a higher temperature, we can suppose that some masses of nonmetallic ingredients in the fluid steel are dissolved at higher temperature and that their precipitation is very limited. Of course the degree of solution depends upon the overheating of steel in the furnace.

The statement that steels made under white slag contain 25 to 50 per cent less oxygen was based on analyses taken from slowly-cooled samples. The addition of 0.1 per cent silicon or 0.25 per cent aluminum to the steel at the time of the taking off of the slag, insures a low initial oxygen content but only as long as this element exists in the fluid steel. We know, however, that in the case of a basic procedure, silicon is burnt out very fast and the equilibrium atmosphere-bath, introduces oxygen again. In practice, this method means that the refining period starts with a low oxygen content, which is of course very desirable, but that it finishes with a higher one. If 0.1 per cent silicon is kept constantly from start to finish, the oxygen content will be sure to be kept on the lowest possible level.

As far as the addition of aluminum is concerned, many steel-makers

are of the opinion that in the case of steels tested by transverse tests, this element represents a harmful factor. The writer agrees with this opinion entirely as he had very sad experiences in this respect. As for grain size specifications, the writer believes that the value of this problem was more exaggerated by theorists than really found in daily practice. It appears that in many cases where grain size was specified before the war, the metallurgists now prefer a coarser grain. In the case of alloy steels, the beneficial addition of aluminum was never sufficiently proved as it was darkened by the influence of alloy elements. When this continent faced a shortage of aluminum two years ago, some articles were published in the technical press, proving that shell steel (carbon steel) should not be fine-grained. On the contrary, in the case of bigger shells, the coarse-grained steel was desirable because of better hardenability. We do not neglect the importance of this problem as one which threw much light on our common knowledge of the life structure of steel; we also agree that for certain tool steels, and for low carbon carburized steels, this is an indispensable factor, but it seems to us that many people started to operate with grain specifications when conditions did not require them at all, or required contrary specifications.

The problem is, what will be cheaper? To apply a little higher silicon content and additional heat treatment, in order to avoid brittleness, and to have lower conditioning costs and higher ingot yield; or to have lower silicon (which does not exclude brittleness), higher billet and bar conditioning costs and lower ingot yield? The answer should be found by practice.

Replying to Mr. W. J. Reagan's discussion; first of all, the writer did not intend to suggest any steel-making procedure. This paper is the comparison of three different deoxidation methods and their influence on physical properties in longitudinal and transverse directions. Finding that one was better than the remaining two, we looked for the reasons and endeavored to explain them using essential data more or less known in the technical literature. The writer did not introduce anything new in steel-making. These three methods are widely used by almost all of U. S. A. steel-makers and the writer never heard of "entirely different practices" as Mr. Reagan says. However, he would be grateful to Mr. Reagan if he would furnish data on steel-making which would basically differ as far as solid scrap charge is concerned.

Some of the physical properties obtained almost constantly when using the white slag method are as follows:

The tests taken from a 10-inch forging (forged from 22-inch ingot) on a 2-inch radius from the center of the forging show, after heat treatment, the following results in transverse direction:

Yield	65 to 67 Tons
Elongation	16 to 20 Per Cent
Reduction of area	40 to 50 Per Cent

The type of steel is similar to that mentioned in this paper.

The writer believes that until a steel-maker goes through the hard school of making steel from which transverse tests are taken, he is not aware of one more difficulty in steel-making.

It seems to the writer that by denying the importance of the manganese-silicon ratio in the charge, Mr. Reagan touched the most important and

essential point of steel-making in any furnace. It could be thought from his statement, that steel-makers should not pay any attention to the charge and particularly to the manganese content on the first test. It is very difficult to discuss this point here. We only quote such famous investigations as those of Herty, Körber, and Olsner, etc. These are, we believe, fundamental works which brought most beneficial indications to steel-making. On the strength of these investigations, the silico-manganese and its chemical composition appeared in steel-making. Almost every steel-maker knows that before adding silicon, manganese should be added to the aims. All these procedures and investigations claim a certain manganese-silicon ratio in any stage of steel-making. When the charge contains about 0.3 per cent silicon, it seems that for high quality steels, all efforts should be made to create fluid products as a result of silicon oxidation during melting.

In the course of his practice, the writer learned of a few plants where electric furnaces made 8 to 10 heats of straight carbon commercial steel a day. The output was the most important thing, as they were supposed to compete with open-hearth furnaces. They met many difficulties in rolling when the charge was melted with low manganese. Later addition of manganese did not improve matters very much. Some heats cracked in roughers on first or second passes. When they started to add higher manganese to the charge, the rolling difficulties disappeared almost entirely. Analyses of inclusions on cracked heats showed that SiO_2 was the dominating ingredient (over 40 per cent). In other words, there were no conditions to create fluid silicates which, of course, should rise up very quickly during the melting period. Analysis of the inclusions after the addition of manganese to the charge was applied and showed an SiO_2 content below 20 per cent. It seems to the writer that the importance of manganese-silicon ratio in the charge should not be ignored. A high manganese content is not an indication of clean steel but it is one of the most important factors to make clean steel.

This point was explained in reply to Mr. Soler's question.

We agree with Mr. Marsh's thesis and there is nothing in this paper in contradiction to this thesis. Mr. Marsh presented the influence of carbon content on oxygen content in steel. We also discussed deoxidation conditions where carbon is almost constant. These are two quite different problems and we do not understand the reason of Mr. Reagan's discussion regarding oxygen.

The author agrees with Mr. Reagan that we do not know as yet whether 0.005 or 0.01 per cent of oxygen is beneficial for high quality steels. It seems however that as far as physical properties in transverse direction are concerned, highly deoxidized steel gives the best results. Nevertheless, it does not mean that in all cases where transverse physicals are required, we must use the procedure which gives highly deoxidized steel. On the contrary, in some cases we are compelled to make steel from which transverse tests are required even under black slag.

The author did not intend to talk about output and "records" of output. However, in answer to Mr. Reagan's question concerning the white slag procedure, we may state that the heat lasts about 5 hours. This takes place of course when solid scrap charge is used.

A HARDENABILITY TEST FOR LOW CARBON AND SHALLOW HARDENING STEELS

By O. W. McMULLAN

Abstract

The 1-inch diameter solid cylinder end-quenched has become widely accepted as the standard method for determining hardenability of medium carbon alloy steels. The variety of methods in use for shallow hardening carbon steels and carburizing steels of all grades indicates that no one method in current use has been found to be entirely suitable for testing all these grades.

This paper describes experiments and lists results obtained from jet quenching one face only of tapered or wedge-shaped specimens and taking hardness readings along the other face of the wedge. By taking readings on a face oblique to the quenched face, rather than on one perpendicular to it as in the standard end quench method, the hardened zones are greatly widened. This permits more readings and less error from inaccuracy of spacing. No cutting after hardening is necessary. Consistent results have been obtained on several types of steel.

INTRODUCTION

THE end quench method developed by Jominy and Boegehold (1)¹ has become widely accepted as the standard method for determining hardenability of steels that develop a considerable degree of depth hardness and have higher carbon content than usually found in carburizing steels. The fact that no essential changes have been made in the original specimen and procedure indicates that they were eminently suitable for the purpose intended as applied to alloy steels above 0.25 or 0.30 per cent carbon.

On the other hand the lack of general acceptance of this or any other method for the carburizing grades and for the shallow hardening steels, the latter category embracing most carbon steels, provides evidence that no entirely satisfactory method has been developed for such steels. Jominy's (2) L-type specimen, solid cylin-

¹The figures appearing in parentheses pertain to the references appended to this paper.

A paper presented before the Twenty-sixth Annual Convention of the Society held in Cleveland, October 16 to 20, 1944. The author, O. W. McMullan, was metallurgist, Youngstown Sheet & Tube Co., Indiana Harbor Works, East Chicago, Indiana. Now chief metallurgist, Bower Roller Bearing Co., Detroit. Manuscript received June 15, 1944.

ders, wedge and cone-type specimens have been used but they possess certain disadvantages, some of which are mentioned below.

A fundamental idea of the standard end quench method is a cooling rate produced by conductivity of heat through the metal to a plane surface ideally quenched. The L-type specimen departs from this situation because of quenching one curved surface and a plane surface at approximately right angles. Conduction of heat, therefore, proceeds in more than one direction simultaneously. The abrupt change in section as shown later may result in some lack of differentiation in degree of hardenability. Cracking of the L-type specimen may occur in high carbon steels, and did in the 1080 steel used in experiments described later. The chief objection to the L-type specimen, however, appears to be the difficulty in machining except with good equipment and skilled operators.

The standard solid Jominy bar is being used to a considerable extent for the carburizing grades, but two disadvantages are quite apparent. Many of these steels do not possess sufficient hardenability to give, even at $\frac{1}{16}$ inch, the maximum hardness obtainable from the grade in thin sections. The other disadvantage is that hardness may drop too rapidly to properly space Rockwell readings and accurately follow the change in hardness. For the latter reason the method is not sensitive enough for shallow hardening steels. Readings on high carbon steels may be quite inaccurate because of minor errors in spacing.

To obtain a general hardenability rating from solid bars quenched all over requires more than one size or mathematical calculations that may be rather complicated and are not 100 per cent accurate.

An extension of the fully hardened and transition zones, with consequent avoidance of errors in hardness results from inaccuracy of spacing or too abrupt change in hardness, has been accomplished by the use of wedge or cone-shaped specimens, particularly in testing high carbon steels of shallow hardening characteristics. Unfortunately these specimens or the methods used likewise have disadvantages. The chief disadvantage is that the specimens must be sectioned after quenching in order to determine the extent of the hardened zone. This involves the possibility of affecting the actual hardness, particularly of high carbon steels, as well as time and expense for the work. Methods of avoiding sectioning have been proposed but apparently have not met with wide acceptance.

The method described here combines the desirable features of the end quench and the wedge and at the same time eliminates certain objections to both. It is based on the end quench idea but face quench might be more descriptive. The specimens are tapered or wedge-shaped and quenched on one face only of the wedge in the same manner as used for the end quench. Sectioning after hardening is unnecessary. Hardness readings, instead of being made on a surface perpendicular to the quenched face, are taken along the other face of the wedge forming an angle with the quenched face. By changing the angle the fully hardened and transition zones can be extended along the oblique face on which readings are taken to fit any degree of hardenability within limits of reasonable specimen size. Obviously the fewer the number of standard specimens the better and it appears that not more than two different angles would be required to cover the entire range desired.

EXPERIMENTAL

The first test specimens were made by cutting off an ungula or wedge-shaped section from the end of a standard Jominy bar. These specimens will be referred to as cylindrical wedges to distinguish them from later specimens cut from bars of a rectangular cross section and designated rectangular wedges. The taper of wedges from the Jominy bar was 1 to 1 (angle of cut 45 degrees, tangent 1). Rather than cutting to produce a sharp edge, cylindrical wedges were made $\frac{1}{16}$ inch thick at the minimum dimension and $1\frac{1}{16}$ inch at the back. This was done to avoid too much air cooling at the thin edge during transfer to the quench and at the same time leave the back thick enough to get a zone of low hardness. The center portion of the Jominy bar was then cut away and the head or collar end and the tapered specimen joined by drilling, tapping and inserting a $\frac{1}{4}$ -inch rod in the manner used for the 3-inch Jominy bar. Specimens were heated and end-quenched in the usual manner. This procedure was unsatisfactory because of the effect of the rod in retarding cooling at the center of the specimen. This difficulty was overcome by a simpler method of connecting the head and specimen. A vertical hole $\frac{7}{8}$ inch in diameter was drilled into the specimen at the thick portion of the wedge close to the cylindrical surface and a similar hole in a corresponding location in the head. The two sections were then joined by a straight cold-drawn wire $\frac{1}{8}$ inch in diameter ground

to a slight taper at the ends and pressed into the drilled holes to hold the quenched face and collar parallel and 4 inches apart. This assembly is shown in Fig. 1. Subsequent use of a longer head section and shorter wire was more satisfactory. During heating, samples were hung vertically from the collar through a hole in a small plate elevated above the furnace floor. The specimen itself rested lightly in cast iron chips. Specimens were placed in a furnace at heat and held there for 30 minutes. Because of their small size they appeared to be fully up to heat in 10 minutes. The wire connection proved to be entirely secure in every case and with reasonable care in handling no misalignment occurred and a uniform "umbrella" of water was shed from the end during the quench. No trouble was encountered from too much loss in temperature on the thin edge during transfer to the quench.

A few simple jigs were made to produce and test the specimens. If a saw or cut-off wheel of adjustable angle is not available, a jig may be made by drilling a hole of the proper size, depth and angle in a flat steel block, such as shown in Fig. 2. The turned cylinder can then be inserted, held in place by a set screw and formed to shape by sawing, shaping or grinding as desired. The same jig can be used for grinding to remove decarburization on the surface before testing if a controlled atmosphere furnace is not available. Such a jig is not satisfactory for holding during the Rockwell test since the sample tends to rotate. By grinding a flat on the back of the cylindrical wedge and the use of a V-block with the proper angle of notch, consistent Rockwell readings were obtained. A jig for holding the specimen while grinding this flat was made by drilling a 1-inch hole in a steel block and then cutting away the top to expose part of the hole as shown in Fig. 3. A set screw at the proper angle holds the sample securely.

If volume of production warranted the tool cost the test specimens described above could be produced readily by cutting a gap in a standard 1-inch specimen with a milling cutter having a 45-degree face angle. The cut would not be entirely through, but would leave just enough metal at the back to hold the specimen in alignment but not enough to affect the cooling rate.

Compositions of steels tested are listed in Table I. The carburizing grades were normalized at 1700 degrees Fahr. (925 degrees Cent.) and the higher carbon steels annealed at 1550 degrees Fahr. (845 degrees Cent.) prior to machining specimens. The tempera-

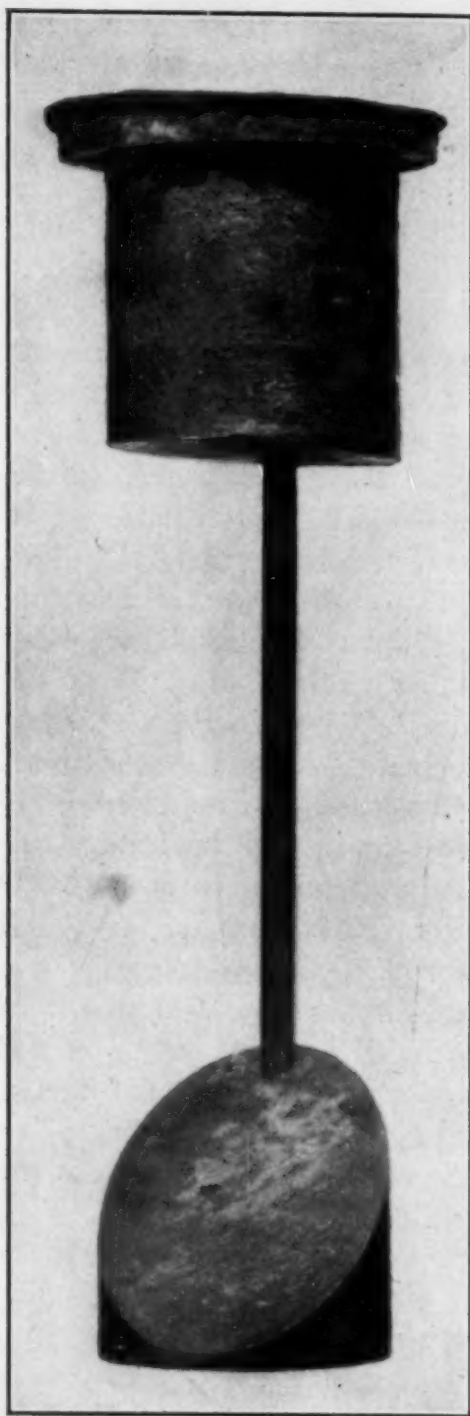


Fig. 1—Cylindrical Wedge Specimen, Angle 45 Degrees, with Assembly for Quenching.

Table I
Analysis of Steels Tested

Steel No.	Type	Bar Size Rounds Inches	Per Cent								Grain Size
			C	Mn	P	S	Si	Ni	Cr	Mo	
1	C-1022	1½	0.22	0.88	0.010	0.022	0.19	7/8
2	C-1040	1¾	0.42	0.79	0.015	0.030	0.19	6/7
3	C-1045	1½	0.48	0.79	0.010	0.025	0.24	6/7
4	C-1045	1¾	0.47	0.92	0.015	0.013	0.28	6/7
5	C-1050	2½	0.51	0.80	0.009	0.030	0.22	6/7
6	C-1080	1½	0.84	0.73	0.009	0.028	0.12	5/6
7	C-1080	1¾	0.83	0.84	0.016	0.026	0.20	6/7
8	C-1095	1½	1.00	0.45	0.025	0.033	0.16	6/7
9	A-4621	1¾	0.18	0.78	0.009	0.015	0.22	1.78	0.23	7/8
10	A-4815	2	0.15	0.49	0.007	0.026	0.25	3.29	0.25	7/8
11	NE-8720	1¾	0.20	0.82	0.011	0.012	0.29	0.50	0.48	0.24	7/8
12	NE-8720	1¾	0.22	0.82	0.009	0.026	0.22	0.55	0.46	0.22	6/8
13	NE-8720	1¾	0.25	0.74	0.010	0.019	0.26	0.49	0.45	0.23	7/8
14	NE-8620	1¾	0.21	0.76	0.013	0.028	0.21	0.53	0.43	0.20	6/8
15	NE-8620	1¾	0.22	0.72	0.011	0.034	0.26	0.53	0.46	0.20	7/8
16	NE-9420	1¾	0.23	0.93	0.007	0.015	0.23	0.43	0.37	0.10	6/8

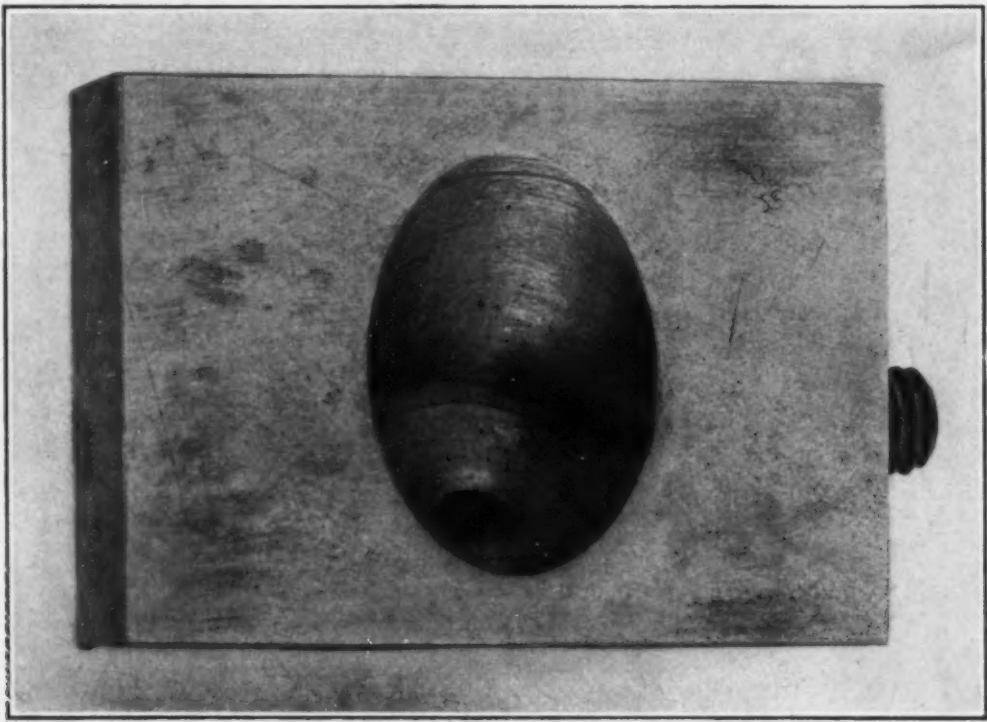


Fig. 2—Jig for Holding Round Bar to Cut Specimen to Shape and for Face Grinding After Quenching.

tures from which these machined test specimens were quenched are shown in the following tabulation :

	Degrees Fahr.	Degrees Cent.
Carburizing grades	1625	885
1040-1050	1525	830
1080, 1095	1475	800

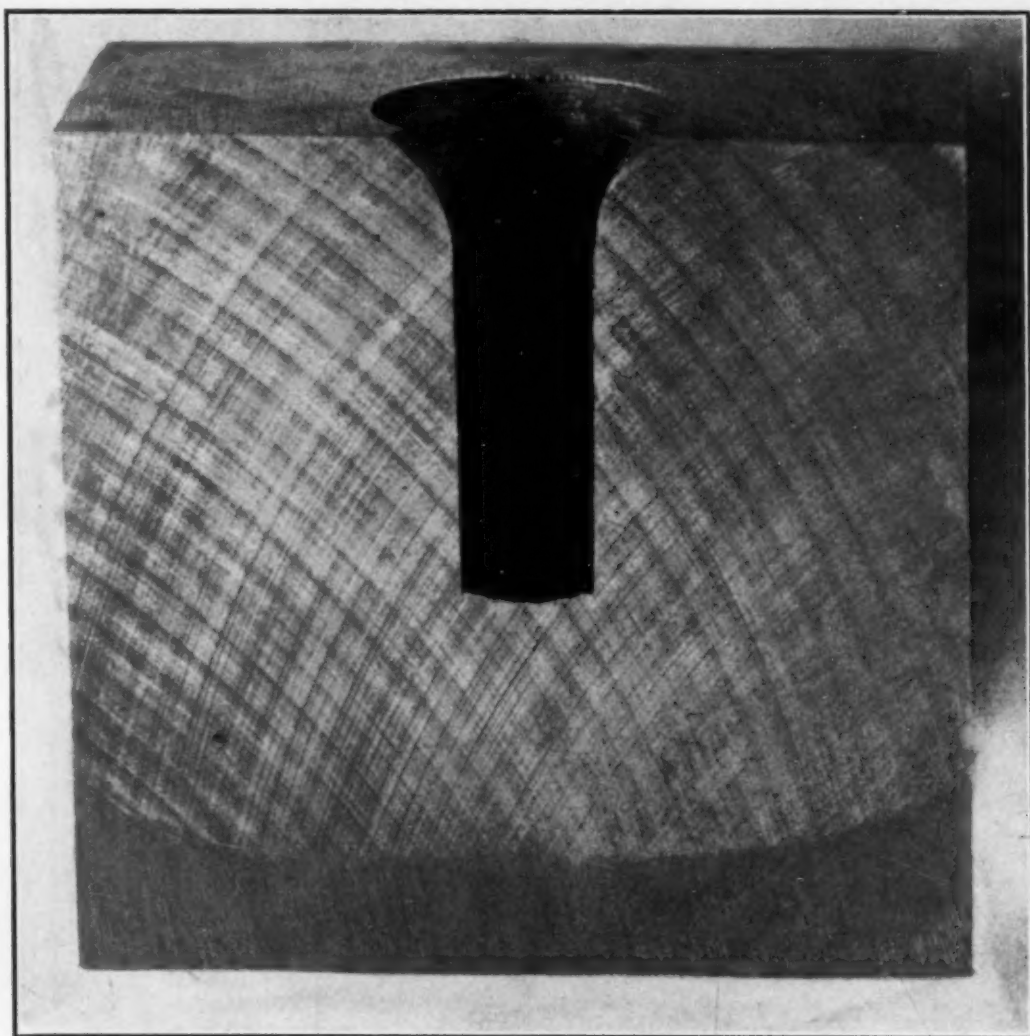


Fig. 3—Jig for Grinding Flat on Back to Facilitate Rockwell Testing.

Quenching conditions were the same as employed for the standard Jominy test. Hardness readings were taken as already described spaced $\frac{1}{16}$ inch apart, the distance being measured from the thin edge of the wedge rather than from the quenched face.

RESULTS

Table II lists results from a number of alloy carburizing steels. The specimens were machined from forged bars on which heat hardenability ratings had been made by the use of the standard solid specimen. Results from both types of specimens are listed for comparison. Table III gives results from several carbon steels of medium and high carbon content.

Table II
Hardness Results from Standard Jominy and 45-Degree Cylindrical Wedge Specimens
Alloy Carburizing Grades

Steel No.	Type	Speci- men	Rockwell "C" at Distance Shown in $\frac{1}{16}$ Inch																
			1	2	3	4	5	6	7	8	9	10	11	12	14	16	20	24	32
9	4621	{45°	44	44	43	42	41	39	37	34	31	29	28	26	24	23	23
		{Std.	43	40	35	32	28	27	25	24	23	23	23	22	21	21	21	18	16
11	8720	{45°	46	46	45	44	43	42	39	37	34	33	31	30	29	28	28
		{Std.	46	45	40	35	33	31	30	29	28	27	26	25	24	23	21	21	19
12	8720	{45°	45	44	43	41	38	35	32	29	28	27	26	25	24	24	25
		{Std.	45	42	38	34	30	29	26	25	25	24	24	23	21	20	19	18	16
13	8720	{45°	50	49	49	48	46	42	38	36	34	33	32	31	28	29	29
		{Std.	45	41	35	32	31	30	28	27	26	26	25	25	24	23	22	21	19
14	8720	{45°	46	45	42	40	36	34	30	29	28	27	26	25	24	25	25
		{Std.	44	42	36	32	27	25	23	22	22	21	20	20	18	17	15	13	11
16	9420	45°	46	46	45	41	38	35	32	31	30	28	27	27	26	26	25

All samples cut from 1-inch rounds turned from center of forged bars approximately $1\frac{1}{4}$ inch diameter.

The results show the anticipated extension of the hardened zone along the sloping face. The specimens are too small to show hardenability in very large sections but the most useful part of the curve follows a consistent pattern that can be duplicated on check specimens. The head or collar end of the specimen, not being quenched, can be used to obtain normalized hardness if desired. In case of the alloy steels there is some indication of the effect of air hardening near the back of the wedge due to the smaller section caused by the cylindrical shape of the wedge. With the 1022 steel the hardened zone is still quite narrow and the curve drops rather abruptly.

To further widen the hardened zone additional samples were cut with taper of 1 to 2 (angle 26.5 degrees, tangent $\frac{1}{2}$). These samples from 1-inch rounds were so small that quick transfer to the quench was required to prevent too much air cooling and also left little or no unhardened portion at the back of the wedge. Rectangu-

Table III
Hardness Results from Standard Jominy and 45-Degree Cylindrical Wedge Specimens
Carbon Steels

Steel No.	Type	Bar Size Rounds Inches	Specimen	Rockwell "C" at Distance Shown in $\frac{1}{16}$ Inch															
				1	2	3	4	5	6	7	8	9	10	11	12	14	16	20	32
8	1095	$1\frac{1}{8}$	45°	67	67	66	60	46	46	46	45	45	44	43	42	40	38	33	..
6	1080	$1\frac{1}{8}$	{45°	66	67	66	65	57	45	45	44	43	42	42	41	40	38
			{Std.	65	64	59	45	..	44	..	44	..	43	..	42	41	40	38	..
5	1050	$2\frac{1}{4}$ *	45°	61	60	58	52	41	34	33	32	31	30	29	28	27	26	23	..
3	1045	$1\frac{1}{4}$	45°	60	59	58	49	41	34	33	33	32	32	31	29	27	25	24	..
2	1040	$1\frac{1}{8}$	45°	58	56	54	47	39	34	33	32	31	30	29	28	26	25	22	..

*Forged bar; others rolled. All specimens cut from 1-inch rounds machined from center of bar sizes listed.

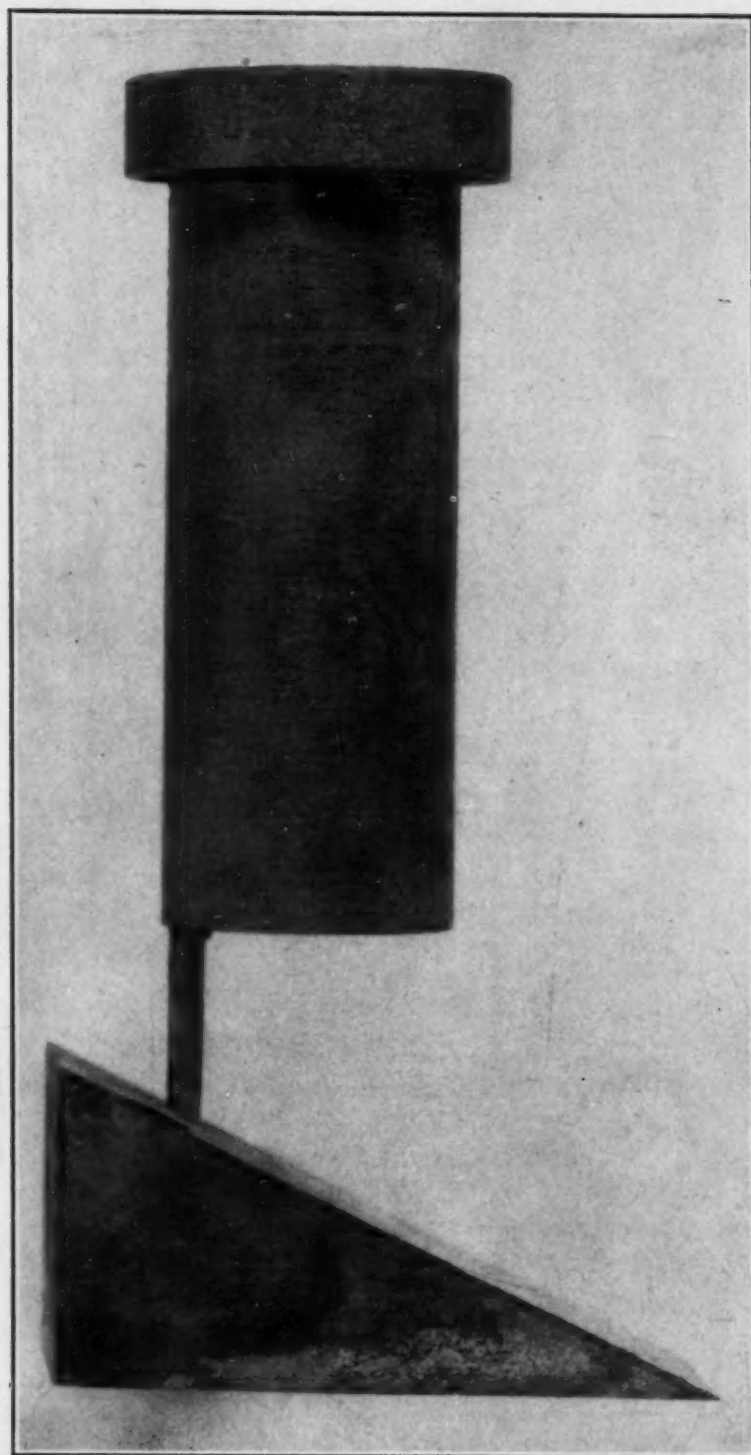


Fig. 4—Rectangular Wedge Specimen, Angle 26.5 Degrees, with Assembly for Quenching.

lar bars 1 inch wide were shaped from rounds and then cut at 26.5 degrees to the axis to produce rectangular wedges 1 inch wide, 2

inches long on the base, 1 inch thick at the back and cut to a sharp edge. To make use of standard Jominy equipment, the assembly shown in Fig. 4 was made for heating and quenching. A standard specimen with collar was cut off to the required length and the specimen to be treated attached with cold-drawn $\frac{1}{8}$ -inch wire as previously described. The face of the specimen and the collar are parallel and 4 inches apart. The $\frac{1}{2}$ -inch round stream was replaced

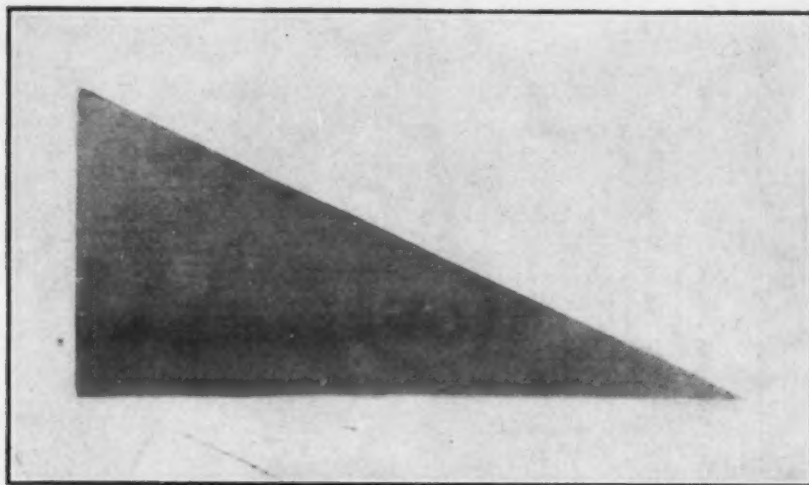


Fig. 5—Longitudinal Section Through Center of Wedge, 1045 Steel, No. 4, Etched to Show Hardened Zone.

by one through a slot 1 inch long by $\frac{7}{16}$ inch wide with $\frac{7}{32}$ -inch radii at the ends. Free height of the water column was 3 inches. Specimens were supported in the furnace as previously described, total time in the furnace being 45 minutes. The water was shed from the rectangular face apparently just as uniformly as from a circular face. The hardened zone of a specimen of 1045 steel, No. 4, is shown in Fig. 5.

Steels 1, 4 and 7 were chosen to represent a carbon series from high to low carbon content and Steels 1, 10 and 15 represent carburizing grades from shallow to rather deep hardenability. Standard Jominy, Type L, 45-degree cylindrical and 26.5-degree rectangular wedge tests were made on adjacent sections of the same bar from each. Results on the wedges are averages of three parallel lines of readings on two or more samples from each steel. Some scatter of results occurred on the weak hardening 1022 steel, but results were as consistent on the wedges as on the standard or Type L specimens.

Results from these five steels are listed in Table IV and plotted in Figs. 6 to 11 inclusive. Figs. 6 to 9 inclusive show curves from

Table IV
Hardness Results from Four Types of Test Specimens. Shallow Hardening and Carburizing Series

Steel No.	Type	Specimen	Rockwell "C" at Distance Shown in $\frac{1}{16}$ Inch																17	18	19	20	22	24	28	32	40	
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16										
1	1022	Standard	38	25	17	13	12	11	8.5	7	..	6	..	6	..	4.5	..	4	2	..	1	0	
		45° C	39	34	26.5	19	16	13.5	12	10	9	8	7	6	..	5.5	..	4.5	..	3	..	2.5		
		Type L	43.5	42	41	38	36.5	35	33	30.5	28	25	23	18	16	14	12	11	..	9	..	7.5	7	5.5	3.5	2	..	
		26- $\frac{1}{2}$ ° R	42	39	36	35	33	29	26	23	21	18	16.5	15	13	12	11	10	..	8.5	..	8	..	7	5.5	5	..	
4	1045	Standard	57	53	37	31	29	28	27	26	..	25	24	..	23	..	22	19	17	16	15	14	
		45° C	61	59.5	58	55	48	39	32	29	27.5	..	26	..	25	24	..	22	..	21	19.5	..	26	23	21	17		
		Type L	63	62	..	62	..	61	60	59	58	56	54	50	48	44	39	34	31	30	..	29	27.5	26	23	21	17	
		26- $\frac{1}{2}$ ° R	..	61	..	61	..	60	59	58	57	55	52	47	40	33	31	29	..	27	..	26	25	24	23	22	..	
7	1080	Standard	67	66	58	45	44	43	..	42	..	41	..	39.5	..	38	36	33	31	29	28
		45° C	66	66	..	66	65.5	64.5	59	46	44	..	43	..	42	..	41	40	39	38	41	38	35	31
		Type L	67	67	66	..	66	..	65	64	56	45	44	..	43	42	40	38	..
		26- $\frac{1}{2}$ ° R	..	65	66	66	..	66	..	65	..	64	63	60	54	46.5	..	44.5	..	43	..	42	40	38	..	
10	4815	Standard	42	42	39	34	30	26.5	25	24.5	..	22	22	21	..	20	..	19	18.5	..	17	16	15	15	
		45° C	42	43	43	41	39	37.5	36	33	31.5	29	28.5	..	27	26	..	26	..	26	
		Type L	44	45	..	45	..	45	..	44	..	43	..	43	..	42	40	39	36	33	30	28	25	22	19	18	16	
		26- $\frac{1}{2}$ ° R	..	43	43.5	44	..	43.5	43	42	41.5	40.5	39	38	37	36	34	32	..	30.5	..	28.5	..	27	25	24	..	
15	8620	Standard	47	45	39.5	33	29	27	26	24	..	23	21	..	19	..	17	16	15	15	14	10	
		45° C	48	47	47	46	45	43	41	38	35	33	32	..	31	..	31	30	30	29	28	27	
		Type L	48	..	48	48	..	47	..	46	45	44	43	41	38	35	32	30.5	29	27	24	22	18	16	12	
		26- $\frac{1}{2}$ ° R	..	47	47.5	..	48	..	47.5	47	46	45	44	42	41	39	37.5	36	34.5	33.5	32.5	31.5	30.5	29	27	25	..	

C—Cylindrical Wedge. R—Rectangular Wedge.

C—Cylindrical Wedge. R—Rectangular Wedge.

the different steels tested by each method whereas Figs. 10 and 11 compare the different methods on the same steel. The curves are largely self-explanatory. Three points are of interest. First it may be noticed on low carbon steels maximum hardness that might be obtained in thin sections is not developed at all in the standard Jominy specimen. The second point, the extension of the hardened zone along the surface tested and more accurate hardenability pattern obtainable, is obvious. The third point involves the comparison of the Type L and 26.5-degree wedge results. Note in Fig. 8 that the slopes of the Type L Jominy curves are of similar degree on the 1045, 8620 and 4815 steels. Obviously the latter two possess deeper hardening characteristics and this is clearly shown by the curves from the wedge specimen, Fig. 9. There is no intention to infer that the Type L curve as a whole does not show the difference between the types of steel since it does so very well but it is believed, on certain types of steel particularly, that the wedge specimen presents a clearer picture of the entire hardenability pattern. The standard Jominy specimen is ideal in that the area of the cross section remains constant. The wedge specimen approaches this condition in that the change in section is uniform, whereas there is an abrupt change in the Type L. This sudden decrease in cooling rate may cause it to fall below the critical rate of several grades of steel within a short section and make the design of the specimen stand out more prominently on the curve than differences in hardenability.

Results from the wedge tests in Figs. 7 and 9 are plotted in distance from the thin edge of the wedge rather than perpendicular distance from the quenched face as is done for the standard Jominy. Results from the various steels have been plotted in distance from the quenched face for all types of specimens. The wall thickness of the Type L specimen was used as the distance until the bottom of the cavity was reached but from there on as distance from the plane through the bottom of the cavity. An unexpected result was that in each type of steel the curves from the 45-degree cylindrical and 26.5-degree rectangular wedges are nearly identical. Whether this is mere coincidence because of specimen size and quenching procedure or has some further significance has not been determined. Curves from the 1045 steel, No. 4, plotted in the above manner are shown in Fig. 12.

The opinion has been expressed (3) that in carburizing grades of steel case hardening characteristics should be determined as well

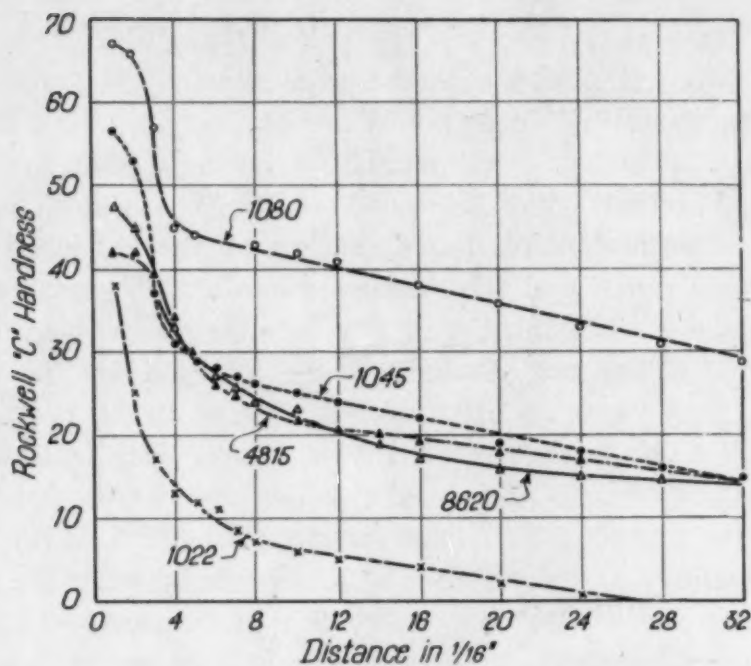


Fig. 6—Standard End Quench Hardness Curves from Steels 1, 4, 7, 10 and 15.

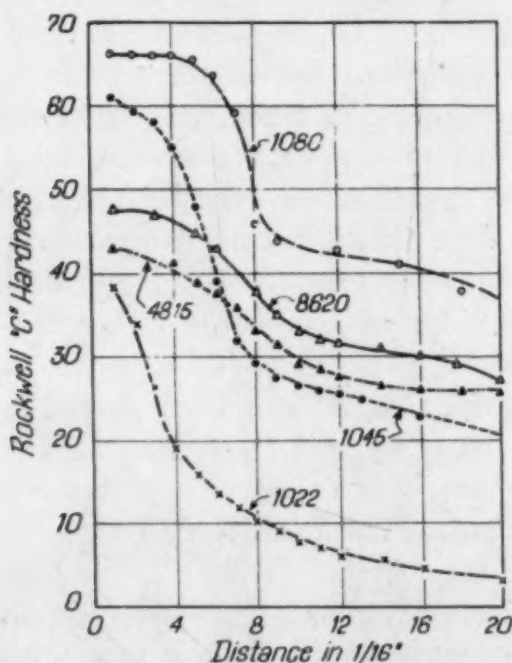


Fig. 7—Hardness Curves from 45-Degree Cylindrical Wedges. Same Five Steels as in Fig. 6.

as those of the core. The standard Jominy bar is well suited for this purpose with alloy steels but has the disadvantage mentioned earlier for the shallow hardening carbon steels. The wedge samples de-

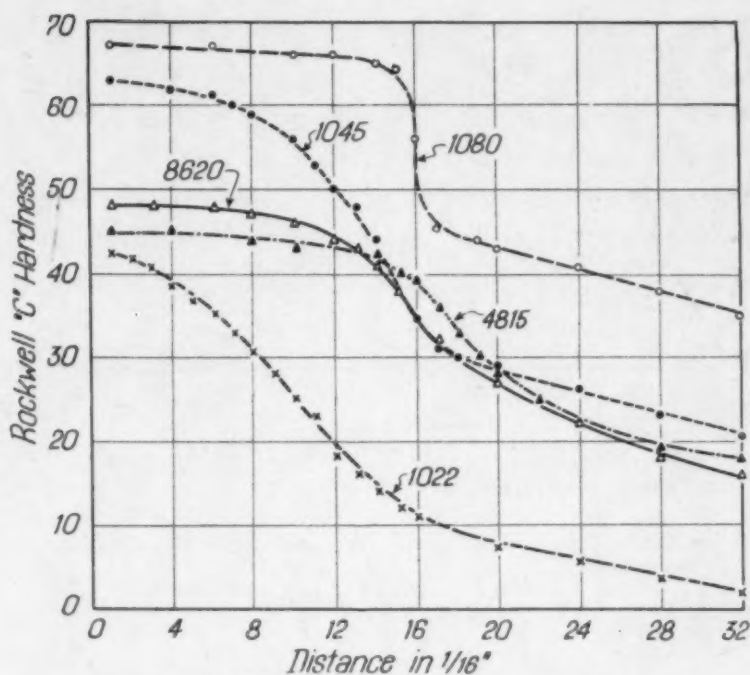


Fig. 8—Type L Hardness Curves. Same Five Steels as in Fig. 6.

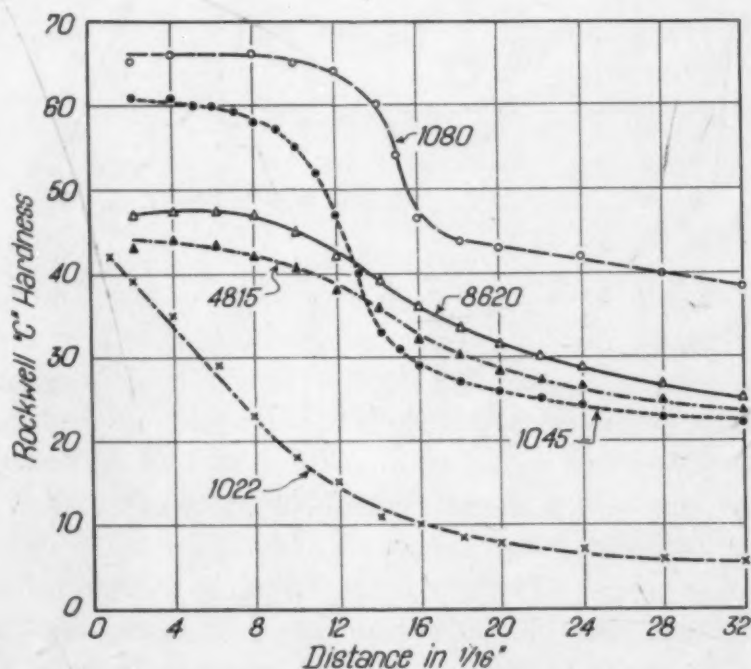


Fig. 9—Hardness Curves from 26.5-Degree Rectangular Wedges. Same Five Steels as in Fig. 6.

scribed should be suitable for the latter since the hardenability of the case should follow a pattern similar to that of the 1080 and 1095 steels.

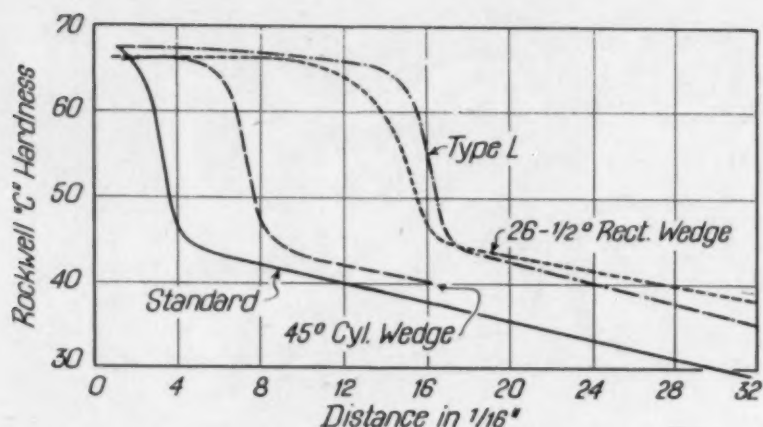


Fig. 10—Hardness Curves of 1080 Steel, No. 7, Obtained from Four Types of Test Specimens.

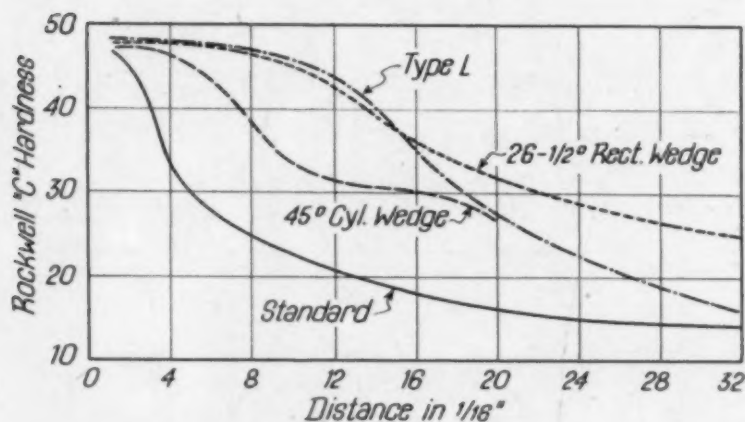


Fig. 11—Hardness Curves of 8620 Steel, No. 15, Obtained from Four Types of Test Specimens.

The writer has been impressed during the course of this work by the remarkable ability of jet quenching one face to give reproducible hardness results. Its development by Jominy and Boegehold was an outstanding contribution to hardenability testing. Morse Hill (4) has determined that in the end quench test the only testing condition which must be rigorously controlled is that there be no scale on the quenched end of the specimen. Not only do a number of variables, within rather wide limits, have little or no effect on standard Jominy results, but the same was found true with a variety of sizes and shapes tested in this work. The essential features are that the steel be at a uniform temperature above its critical range when quenched, and that the quench be quick and completely cover the one face only to be quenched. Tests were made on various bar sizes between $1\frac{7}{8}$ and 2-inch diameter by cutting off slices to form 45 and 26.5-degree cylindrical wedges similar to those from 1-inch

rounds. After grinding the cut faces but without removing scale from the bar surface, these slices were jet-quenched on the circular face and hardness readings taken on the other face of the wedge. Uniformity of results was quite remarkable, even more so than on some of the smaller samples. The procedure offers a quick method of determining differences in hardenability between two or more samples of steel in the same bar size. Results, of course, would be

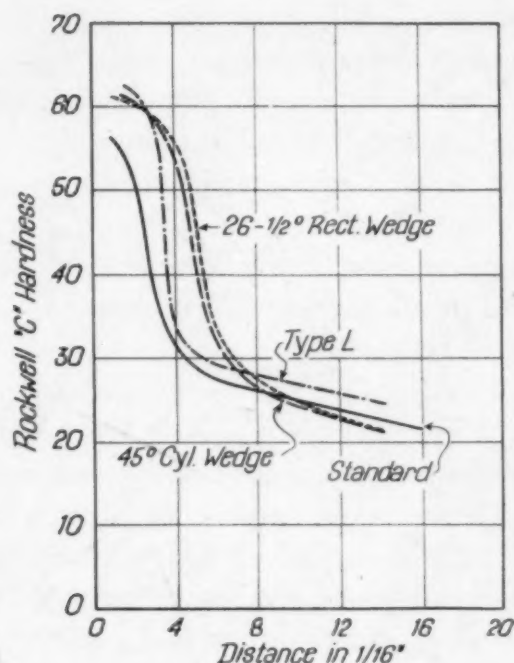


Fig. 12—Hardness Curves of 1045 Steel, No. 4, from Four Types of Test Specimens with Distance Measured Normal to the Quenched Face.

comparative only and not directly convertible to those from a specimen of standard size.

One criticism sometimes made of the standard end quench specimen is that it tests only the outside portion of the bar. The wedge specimens described in these tests check the cross sections and should show any undue segregation if readings are located properly for the purpose.

The writer is well aware that the limited data presented here do not justify the adoption of the method as a production test without further work. Results are presented at this time only because of the need of a more suitable method, particularly in view of the rapid changes and additions to steel compositions. Should

the method prove to be of any value, it is hoped that presentation of the idea at this time, in a more or less rough form, may be justified.

SUMMARY AND CONCLUSIONS

No standard method for testing the hardenability of shallow hardening and low carbon steels has been generally accepted to the extent that the standard Jominy end quench method has for the medium carbon alloy steels.

Various methods in current use possess certain disadvantages. The solid cylinder is not sensitive enough to pick out small differences in hardenability among the shallow hardening steels, the fully hardened zone is too short to give enough readings and the drop in hardness is so rapid that small inaccuracies in spacing readings may cause large errors. It may be unsuitable for carburizing steels for the same reasons and in addition may not show maximum hardness obtainable even at the first reading.

The Jominy L-type bar has not been widely used, probably because of being more difficult to machine. It has a tendency to crack with high carbon steels and the abrupt change in section may tend to cover up small differences in hardenability over a short section. Other methods such as the use of cones and wedges quenched all over involve the disadvantages of sectioning hardened test specimens.

The method described in this paper is a combination of the end quench and wedge-type test. It might be described as a face-quenched wedge since the specimens were wedge-shaped and were quenched on one face only. In this manner, by properly choosing the taper of the wedge, full hardness can be developed in the thin section, the completely hardened and transition zones can be made long enough that inaccuracies from spacing readings and rate of change in hardness are greatly reduced and the hardness pattern more readily followed. Hardness readings were taken along the face of the wedge at an acute angle to the quenched face rather than at a right angle as in the standard end quench test. Sectioning of hardened samples was not required. Thus the fundamental ideas of the end quench and wedge were combined and at the same time certain disadvantages of each were eliminated.

Specimens used in the work were of two designs: Those formed by cutting off the end of a 1-inch round bar at an angle of 45 degrees and those cut from a square or rectangular bar to form a more

acute angle, 26.5 degrees. In order to use the available standard Jominy equipment for quenching, specimens were attached, by means of a small rod, to the collar end of shortened Jominy bars. Heating and quenching procedures used for the end quench were followed except that for the larger rectangular wedges the jet orifice was a slot rather than a round opening. With reasonable care no difficulty in the procedure was encountered and consistent results were obtained.

Several conclusions may be drawn from the work.

1. The hardened zones were widened along the surface tested as anticipated.
2. A satisfactory range of hardness was obtained in alloy carburizing grades from the 1-inch round specimens cut at 45 degrees, tangent 1.
3. Wedge samples cut from rectangular bars were larger and a smaller angle, 26.5 degrees, tangent $\frac{1}{2}$, could be used and thus increase the length of the hardened zones and the total spread of hardness.
4. The mass of hot metal behind the quenched face influences the cooling rate; consequently, readings on tapered specimens are not directly convertible to straight bar results at the same distance from the quenched surface. They can, of course, be converted on the equal cooling rate-equal hardness theory. An advantage of the method, however, is the elongation of the hardness curve. Results, therefore, are plotted to show distance from the thin edge of the wedge rather than from the quenched face.
5. Reproducibility of results by end quench procedure, in spite of a number of variables, was confirmed with specimens of various sizes and shapes.
6. Tapered disks or wedges, produced by merely taking one square cut and one oblique cut across a bar, end-quenched showed surprising uniformity of hardness results in a limited number of tests on several different bar sizes and types of steel. The procedure offers a quick method for comparative purposes.
7. The specimens used in these tests were checked for hardness on the cross section and offer an opportunity for determining uniformity not possessed by some of the other hardenability methods.

ACKNOWLEDGMENTS

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DISCUSSION

Written Discussion: By Chas. H. Shapiro, chief metallurgist, Reed Roller Bit Co., Houston, Texas.

Mr. McMullan, in his development of the tapered-bar or wedge-shaped hardenability test, has made it possible to obtain a greater number of Rockwell readings in the fully hardened zone. In this, he has made a distinct improvement on the standard Jominy bar by arranging the test to be more sensitive to the reading of changes in the zone of high hardness. By his method he has also been able to hurdle some of the disadvantages inherent in the Jominy-type bars, in present use on low hardenability and shallow hardening steels.

The preparation of the sample is, however, more difficult, requiring a number of steps, each of which must be more accurately performed. Considerable care must be exercised during each step of the testing procedure, especially in the hardness reading operation. The fixtures, however, aid materially in simplifying the test piece preparation.

The development of this wedge-type test should result in more accurate determination of hardenability because of the elimination of steep, close-to-vertical drops in the many curves we have had to work with in the past.

Written Discussion: By John G. Kura, research engineer, Battelle Memorial Institute, Columbus, Ohio.

An attempt to use the 45-degree cylindrical wedge test on S.A.E. 1030 steel resulted in a much lower hardness at the first $\frac{1}{8}$ -inch position on the wedge bar than at a similar position on the standard end quench bar. This is reasonable because of the greater distance of this point from the quenched end of the wedge as compared to the standard bar if one disregards the effect of

the greater mass of the standard bar. However, the author indicates that at least the same hardness was obtained at this position on the wedge as was obtained on the standard bar. Was this a general observation regardless of the type of steel?

If the data for the 45-degree cylindrical wedges were to be plotted in terms of the distance from the quenched end and a comparison made with the standard end quench curves, the effect of the difference in mass would readily be seen to be considerable as in Fig. 12. This comparison, of course, is extreme. Yet, in shallow hardening steels, small variations in the length of the wedge cylinders may appreciably affect the results. A study of small differences in specimen dimensions should be undertaken to assure that grinding off a suspected decarburized layer, prior to taking hardness readings, will not unduly affect the results.

To check the results of spacing hardness readings on the standard bar tested in the normal manner, a 45-degree cylindrical wedge was cut off the standard bar of an S.A.E. 1030 steel. Readings were taken on the 45-degree face and plotted in terms of the distance from the quenched end. Practically identical curves were obtained from the 45-degree face and the standard bar indicating that normal, careful spacing of hardness readings on the standard bar are reliable even on shallow hardening steel.

Concerning the fourth conclusion drawn by the author, a plot of the distance from the quenched end of the standard end quench bar versus the distance from the thin edge of the 45-degree cylindrical wedge bar for the occurrence of equivalent hardness shows a distinctly different curve for each of the steels. If the tapered specimens are convertible to straight-bar results on the equal cooling rate—equal hardness theory, identical curves should have been obtained in each case. Some unknown factors appear to be exerting a considerable effect to cause such a discrepancy in duplicating cooling rates.

Written Discussion: By W. E. Jominy, chief metallurgist, Dodge Chicago Plant, Division of Chrysler Corp., Chicago.

I have read the paper by Mr. McMullan with a good deal of interest. It represents an ingenious method for using the end-cooled principle of measuring hardenability. I see nothing fundamentally wrong in the test which Mr. McMullan has devised and whether or not this test is used will depend, I believe, upon the convenience of it. It would seem to me to be a little flimsy to handle when hot and also to require a special fixture to get accurate hardness tests on the piece.

There are certain references made to the L-bar which I should like to discuss. First the matter of the tendency to crack, especially with high carbon steels. In my experience with certain steels, cracks have developed which have been circumferential. In every case, however, the crack has not interfered with the cooling, since it occurs too late to affect this, or with the measuring of the hardness in the fixture. I have not found that the cracking interfered in any way with the test except perhaps to vitiate one hardness reading. In general, the cracking occurs on steels which are deep hardening and which would be better measured with the standard end-cooled bar.

Second, the abrupt change in section referred to in this paper occurs at 1 inch from the end of the L-bar. At this point the cooling rate is 54 degrees

per second at 1300 degrees and it is beyond this point where the change might be considered abrupt. The change in cooling rate from 1 to $1\frac{1}{4}$ inch represents a drop of 28 degrees per second which is a smaller change than occurs in any $\frac{1}{4}$ -inch distance along the specimen to this point. Changes occurring beyond the $1\frac{1}{4}$ -inch distance are still less but would more conveniently be measured on the standard end-cooled bar since this point, namely $1\frac{1}{4}$ inch, corresponds to about $\frac{1}{8}$ inch on the standard bar.

The L-bar was developed mainly for steels which do not harden up for a distance of $\frac{1}{8}$ inch on the standard end-cooled bar and for such steels there is no difficulty about the change which occurs beyond 1 or $1\frac{1}{4}$ inch on the L-bar.

The standard bar will always be used in preference to the L-bar wherever it applies, since it is less difficult to machine.

In our shop the cupped end of the L-bar is machined in about 10 minutes. This is without any special tools. It is possible to make a special reaming cutter to finish the cupped end of the L-bar and with such a cutter the average time for making the cupped end is $3\frac{1}{2}$ minutes. This does not seem to offer too much difficulty.

Written Discussion: By N. E. Rothenthaler and F. C. Young, metallurgists, Ford Motor Co., Dearborn, Mich.

Mr. McMullan's paper on this important subject has been studied with great interest by the writer. There is a crying need for a more simple method of preparing hardenability specimens from shallow hardening steels, for the specimens now in use present complicated machining problems of either preparing the test specimens or sectioning them after quenching. Mr. McMullan has attempted to solve these two problems by his new proposed method. However, he has maintained the basic principles of the standard 1-inch Jominy hardenability test, namely, quenching one face and measuring the differential hardnesses of another face. Also, with a few modifications the present hardenability quenching equipment could be made interchangeable with the 1-inch Jominy bar and this new proposed method.

The application of a 26.5-degree rectangular wedge as illustrated by Mr. McMullan appears to give the closest similarity of curves to the present standard L-type bar. This he has illustrated in Figs. 10 and 11. The present information on file with the L-type bar will not become obsolete and can still be used for reference. However, according to Fig. 11, the rectangular wedge will give a wider spread in the hardening area when measured normal to the quenched surface. This appears not to be affected by the angle of slope or shape of the specimens. We wonder how Mr. McMullan accounts for this condition as there is, no doubt, a different cooling rate with the two different slopes.

This simple type of specimen will overcome a good deal of the hesitancy in obtaining and accepting the hardenability data on the shallow hardening steels. Instead of the 1-inch standard Jominy bar which had been used in preference to the L-type bars for the carburizing grades, we could apply this wedge method and get a greater detail of their hardening characteristics.

In widening the hardening zones for these shallow hardening steels, it is believed greater satisfaction will be felt in the use of the hardenability data.

This wedge method of hardenability testing appears to be both rapid and reliable for reproducibility. The specimens can be easily machined without the use of a skilled machinist. Simple jigs and fixtures can be readily made for production checks.

The present tendency in steel specifications is to include the use of hardenability as a standard range as well as the chemistry with emphasis on the hardenability characteristics of a heat rather than its analysis. Consequently, any means which will promote the efficiency of testing the low carbon steels is to be encouraged.

As Mr. McMullan indicates, more extensive data must be acquired before adopting his proposed method for production use. We sincerely hope he will present his present data along with any additional information to the hardenability committee of the Society of Automotive Engineers. This committee is now engaged in revising the hardenability procedures and consequently would be much interested in this new method of hardenability tests for low carbon and shallow hardening steels.

Written Discussion: By H. B. Knowlton, materials engineer, International Harvester Co., Chicago.

Mr. McMullan has made a valuable contribution to the technique of measuring the hardenability of shallow hardening steels.

The hardness at $\frac{1}{8}$ inch from the quenched end of the standard Jominy specimens seems to be erratic. All efforts to determine hardness at distances closer to the quenched end than $\frac{1}{8}$ inch have been unsatisfactory. Consequently the proposed method offers considerable promise for the measurement of hardenability of the extremely shallow hardening steels.

There are, however, two possible objectives to McMullan's method:

1. If the important part of the hardenability curve involves readings taken near the center of the cross section it would be expected that any segregation of carbon in the center of the bar would produce a considerable error, indicating higher than actual hardenability.
2. The specimen is somewhat more complicated to make than the standard Jominy specimen, consequently should be used only where the simpler specimen is not satisfactory.

This test may also be used to advantage where there is a break in the hardenability curve between the $\frac{1}{8}$ and $\frac{1}{4}$ points on the standard Jominy curve. The same can be accomplished, however, by taking readings on the standard Jominy bar at increments of distance from the quenched end of less than $\frac{1}{8}$ inch. It is not practicable to take readings closer together than $\frac{1}{8}$ inch, but it is possible to take four sets of readings on the standard Jominy bar with the starting point for each series $\frac{1}{8}$ inch farther from the quenched end than the preceding series.

Thus the distances from the quenched end in the first series of readings would be $\frac{1}{8}$, $\frac{1}{4}$, $\frac{3}{8}$, $\frac{1}{2}$ inch, etc., while the second series would be $\frac{5}{8}$, $\frac{3}{4}$, $\frac{7}{8}$, 1 inch, etc. To accomplish this it is necessary to use a fixture which will index $\frac{1}{8}$ inch with reasonable accuracy. The writer and his associates have used a fixture in which the block supporting the specimen is moved horizontally by a screw having a $\frac{3}{8}$ -inch NC thread. One revolution advances the block $\frac{1}{8}$ inch. Quarter revolutions advance the block $\frac{1}{16}$ inch.

It has been found necessary to use a hardened block to support the specimen and to make one Rockwell impression to seat the specimen on the block before taking any actual readings. We have found no error in using cylindrically ground specimens supported on hardened "V" blocks in place of the specimen with the conventional flat ground sides.

If hardenability tests are to be used for acceptance and rejection of incoming heats of steel it would be desirable to standardize on a specimen which can be most readily manufactured. Centerless ground cylindrical specimens would have an advantage in this respect. Both the McMullan specimen and the Type L bar appear satisfactory for measurement of hardenability of very shallow hardening steels. The choice may depend upon the relative ease of preparation of the specimens.

Author's Reply

Mr. Jominy's interest in the paper is appreciated. His comment on the design being flimsy to handle is quite correct as to Fig. 1 but a shorter connecting wire used later as in Fig. 4 eliminated any trouble when the specimen was supported properly in the furnace and handled with reasonable care.

In reference to the abrupt change in section of test specimens, I am of the opinion that the rate of change must be considered in connection with the critical cooling rate of the steel being tested rather than as the amount of drop on the standard cooling rate curve. As shown in Fig. 8 the critical zone for 1080 steel coincided with the change in section at the base of the hole in the L-bar making the hardness curve drop very rapidly whereas with 4815 steel, slower to transform, the curve was not so strongly affected by the change in cooling rate at that portion of the specimen.

It may be observed from Fig. 6 that the hardness of the 1080 steel has started to drop at the $\frac{1}{8}$ -inch position on the standard Jominy bar and beyond this point drops rapidly. A more sensitive test would be one in which the change in cooling rate with distance on the specimen is more gradual at the critical cooling rate of the steel and it is believed that the wedge specimens meet that requirement for certain types of steel better than either the standard or Type L bar.

Mr. Rothenthaler has emphasized the need of a more suitable method for determining the hardenability of shallow hardening steels and the writer is pleased to note that he believes the suggested face-quenched wedge specimen would meet the requirements. Apparently his reference to Fig. 11 should have been to Fig. 12 in connection with the hardened distance measured normal to the quenched surface. Such a comparison between the L-bar and the 26.5-degree wedge can be only a rather rough one since in the L-bar the quenched surface is the inside of the hole, smaller in area than the outside. Thus each unit of surface quenched must remove heat from a larger area back of it instead of a constant area as in the wedge. Also near the bottom of the hole the cooling rate and hardness may be affected by the large mass of hot metal in the solid portion. For these reasons it may be expected that hardness would drop faster in the Type L bar than in the 26.5-degree wedge when plotted in the manner shown in Fig. 12.

Mr. Shapiro has pointed out some of the advantages and disadvantages of the proposed method. More steps may be involved in preparing the sample, but on the other hand, the machining operations are less subject to error than on some other types of specimens. The greatest possibility for error lies in holding the specimen when taking hardness readings.

In answer to Mr. Kura it was found, regardless of type of steel, that the hardness at the first reading of the 45-degree wedge was equal to or greater than the first reading on the standard bar. I see no reason why this should not apply to 1030 also. While it is true that the distance from the quenched face is less on the standard bar, the greater mass of that bar retards the cooling more. In one or two exceptions, disregarded because of excessive delay in getting the specimen to the quench, low readings were obtained at the edge because of temperature drop in the thin section. The time interval would be shorter with a carbon steel, such as 1030, than with an alloy steel with a lower critical point.

Mr. Kura also points out the possibility of variations in mass which would affect results. Two things are important; one that the angle of the wedge be accurate and the other that the respective readings be located at equal distances from the quenched face in all specimens. Change in length of the full cylindrical portion of the 45-degree wedge would affect results. Grinding off the sharp end of the 26.5-degree wedge, such as to remove decarburization, would, because of the taper, change the point of origin of measurement and change the position of all the readings as well as the first. This could be avoided by a slight change in design to leave a blunt end so that, for example, the first reading taken would correspond to the number 2 position reported, which in most cases is the first reliable one because of the thin section near the sharp edge. Removal of the first $\frac{1}{8}$ inch of the sharp edge should have no measurable influence on the hardness pattern in the specimen; it is merely a question of point of origin for locating hardness readings. Provided the angle was kept constant it was found that the overall length of the 26.5-degree wedge could vary considerably without appreciable effect on the results, the action being similar to that of changing the length of the solid cylinder.

Knowledge of hardenability testing has not reached the stage where exact duplication of results by conversion from different types of specimens can be expected based on the equal cooling rate—equal hardness theory. This can be demonstrated readily by attempting to convert standard Jominy results to those obtained from all-over bar quenching. Lack of agreement will be found between standard and L-bar results when compared on the basis of equal cooling rates. Table IV shows at least equally as good agreement between standard bar and 26.5-degree wedges as between standard bar and Type L results. Plotting results from Table IV as suggested by Mr. Kura shows quite good agreement between the 45 and 26.5-degree wedges; much better than between standard Jominy and Type L bars or any other pair among the four methods. I agree that unknown factors are exerting a considerable effect in causing discrepancies. If the equal cooling rate—equal hardness theory is rigidly valid, exact hardness is much easier to measure and it would appear that the error is much more apt to be in duplicating the cooling rate and perhaps even more so in measuring just what the cooling rate is, especially at the more rapid rates.

Mr. Knowlton has brought up some interesting points on hardenability testing. Among others he has raised two possible objections to the proposed method. In answer to the first objection, it is not necessary to take readings at the center of the cross section; in fact, all results reported in the paper were averages of three parallel lines of readings only one of which passed through the center. It was found that readings taken close to the edge of rectangular wedges were 1 to 3 points higher, probably due to air hardening at the corners, than points between (along the same distance line) where uniform cooling took place. Rectangular wedges of the size described cut from the smallest possible rounds provided accurate readings from center to half radius or farther. In case of cylindrical wedges a wider spread was possible but not desirable because of excessive grinding for a wider flat on the back. Therefore, center segregation need have no influence on the test but the method does have the advantage of being able to show center segregation if locations of readings are chosen to do so.

Proposed test specimens are not as simply produced as turning a solid cylinder. Obviously the angle must be accurate but some of the other dimensions are not critical just as the length of the standard Jominy bar is not critical. Further knowledge of the accuracy and finish desired might simplify machining.

Mr. Knowlton suggests the procedure of taking readings, by staggering, at $\frac{1}{8}$ -inch intervals when the break in the hardness curve occurs between the $\frac{1}{8}$ and $\frac{1}{4}$ -inch points. In addition to careful manipulation of accurate equipment the success of such a procedure presupposes an absolutely perfect hardness contour around the specimen. Small ripples in the contour would affect readings at $\frac{1}{8}$ -inch intervals. It has been my experience that variations do exist around the circumference at a given distance from the quenched end, especially in shallow hardening steels where the break in the curve is sharp.

Actual measurement of a diamond impression at 60 Rc shows it to be about 0.020 inch in diameter at the top, and, of course, softer material would have a larger impression. The drop in hardness across the impression itself is therefore greater than between the $\frac{1}{8}$ -inch intervals at which readings are desired. The best that can be hoped is that the reading represents the average hardness over a distance more or less greater than $\frac{1}{8}$ inch depending on the hardness level. I believe, therefore, that widening the hardened zone which decreases the rate of hardness change offers the better possibility of determining accurately differences in hardenability of the shallow hardening steels. The small angle wedge effectively widens the hardened zone not only in the extremely shallow hardening steels but also in those of greater depth hardening which tend to show a steep drop in the standard hardness curve.

DIFFUSION IN POWDERED METALS

By P. W. SELWOOD AND JANE NASH

Abstract

This paper discusses a magnetic method developed by the authors based on the analysis of magnetization temperature curves and used to follow the approach to homogeneity in copper-nickel mixtures. Powdered nickel and copper were prepared by hydrogen reduction of the chemically pure oxides and compacted into pellets. These were vacuum-sealed in glass capsules and suspended from an analytical balance by a platinum wire so placed that they were in the vertical field gradient of an electromagnet. Magnetization and temperature were recorded. While the results obtained in these studies are not entirely perfect it is believed that this method will prove to be a useful tool in powder metallurgy.

THERE is no completely satisfactory method for measuring the rate of diffusion and the approach to homogeneity in a mixture of powdered metals. Definite progress in this direction has been made by Rhines and Colton¹ who used an electrical method to measure the degree of inhomogeneity of a solid solution alloy. The present paper describes a magnetic method which gives a rough quantitative approach to this problem. The system studied was copper-nickel.

It is well known that ferromagnetic substances, at the Curie point, show an abrupt diminution of specific magnetization. For nickel this occurs in the neighborhood of 358 degrees Cent. (675 degrees Fahr.). For homogeneous nickel-copper alloys, and for certain other systems, Marian² has shown that both the magnetic moment of the nickel and the Curie temperature are linear functions of the atomic concentration of the nickel. Copper is, of course, diamagnetic.

In a mixture of powdered metals we have, before sintering, simply a mechanical juxtaposition of the pure components. As

¹Rhines and Colton, "Homogenization of Copper-Nickel Powder Alloys, p. 67, Powder Metallurgy, edited by John Wulff, published by the American Society for Metals, Cleveland, Ohio, 1942.

²Marian, Journal phys. radium 8, 313 (1937); Ann. phys. 7, 459 (1937).

Of the authors, P. W. Selwood is associate professor of chemistry, Northwestern University, Evanston, Illinois, and Jane Nash is connected with the department of chemistry of the same institution.

sintering proceeds, diffusion takes place, and we have a continuous series of solid solutions. Finally, with the approach to homogeneity, the composition becomes uniform throughout. The continuous series of solid solutions has been called by Rhines and Colton a "micro-inhomogeneity." In an earlier paper, Morris and Selwood³ referred to the same thing as "non-equilibrium solid solutions."

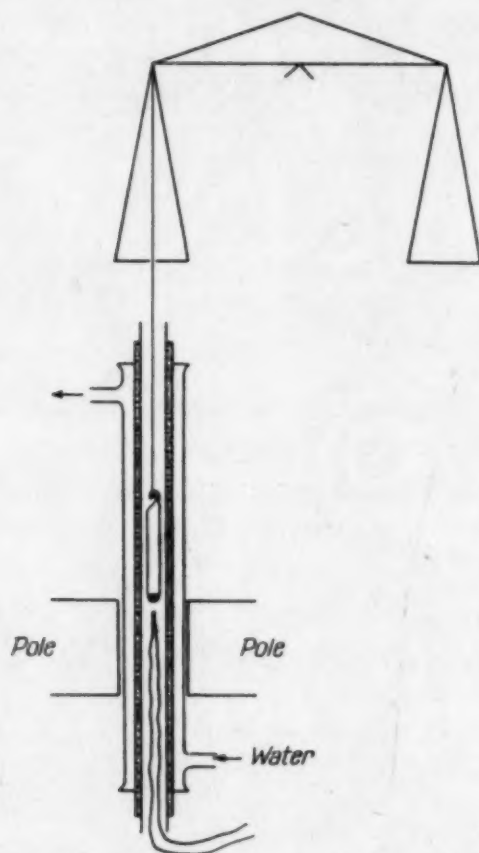


Fig. 1—Balance and Oven for Measuring Specific Magnetization Over a Range of Temperature.

Each concentration represented in the partially homogenized mass has a definite Curie temperature. The magnetization-temperature curves obtained from such systems are therefore the summation of an infinite number of curves, each of which shows a sharply defined Curie temperature. Instead of a single Curie temperature these alloys will show a gradual diminution of magnetization over a wide range of temperature. The rate of approach to homogeneity, the degree of homogeneity, and the distribution of concentrations within the system will all be indicated by the changing slope of the magnetization-temperature curve as sintering proceeds.

³Morris and Selwood, *Journal, American Chemical Society*, Vol. 65, 1943, p. 2245.

EXPERIMENTAL METHOD

Powdered nickel and copper were prepared by hydrogen reduction of the chemically pure oxides. The powders averaged about 300 mesh, but no special pains were taken to insure uniform size. Mixtures of several concentrations were prepared by weighing and thorough stirring. The mixtures were then compacted into pellets under a pressure of approximately 150,000 pounds per square inch.

These pellets were vacuum-sealed in glass capsules then suspended from an analytical balance by a platinum wire. As shown in Fig. 1 the pellets occupied a position inside a heating tube and were so placed that they were in the vertical field gradient of an electromagnet. As the field was applied the samples were attracted toward the center of the magnet with a force equivalent to several tenths of a gram. The field used was about 5000 gauss, enough to insure saturation. The temperatures were measured with a chromel-alumel thermocouple.

No special virtue is claimed for this method. The apparatus happened to be readily available and easy to operate. At the present time an induction apparatus is under construction for further work on this and related problems.

RESULTS

The specific magnetizations over the temperature range 25 to 400 degrees Cent. are shown in Figs. 2 and 3. These are for two representative concentrations: 90 per cent nickel-10 per cent copper and 70 per cent nickel-30 per cent copper. The quantity σ/σ_0 is the ratio of specific magnetizations observed, and for an equivalent weight of pure nickel. Data are shown for the compacted but not sintered samples, and for various stages of heating.

It is clear from these figures that the magnetization-temperature curves change as predicted, and that the approach to homogeneity is indicated by a sharpening of the Curie temperature. Unfortunately for the quantitative interpretation of the results, the Curie temperature of pure nickel itself is not very sharply defined. This difficulty does not, however, prevent some useful conclusions being derived from the data.

Let us consider a partially sintered alloy which gives a magnetization-temperature curve as shown in Fig. 4. Take two temperatures

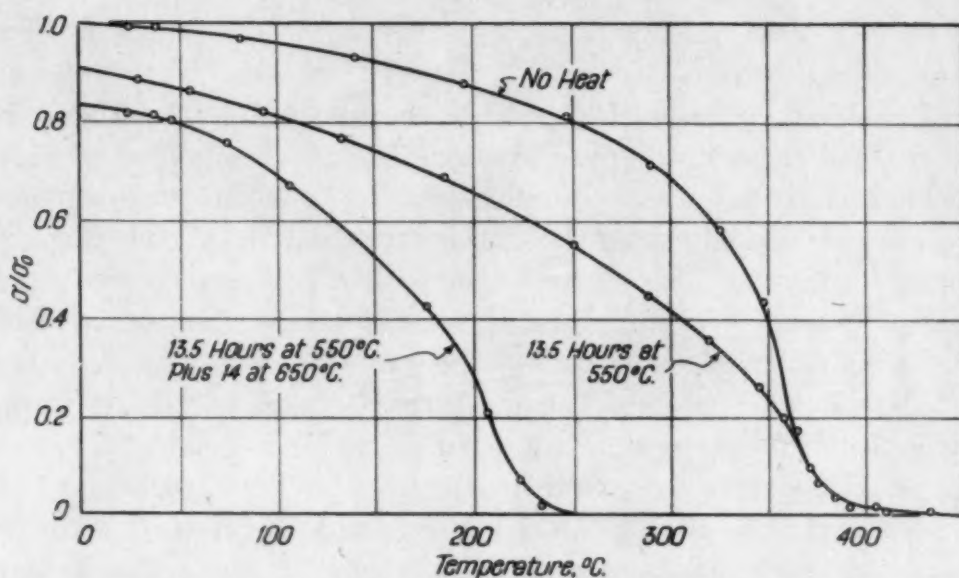


Fig. 2—Magnetization-Temperature Curves for a 90 Per Cent Nickel-10 Per Cent Copper Powder Mixture, Before Heating, and After Two Stages of Sintering.

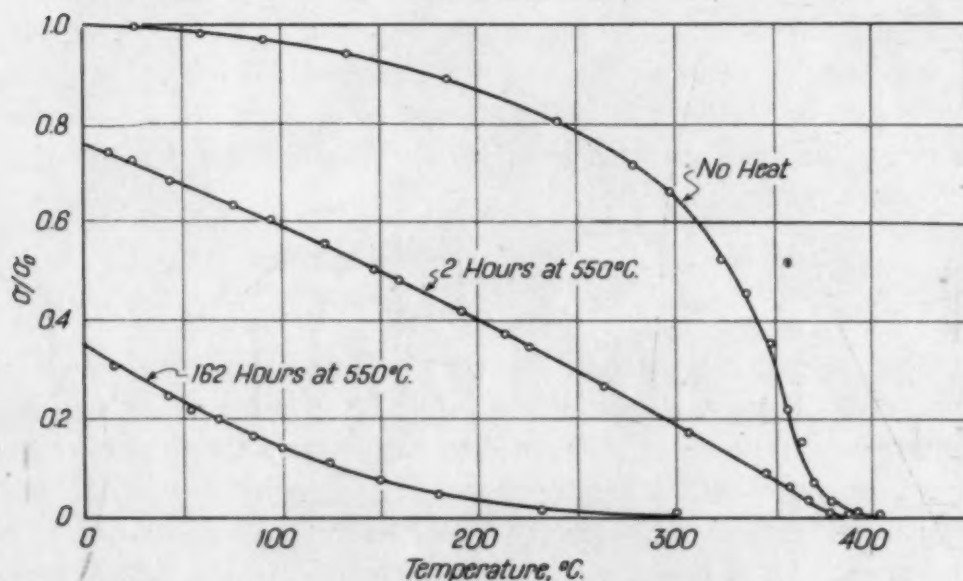


Fig. 3—Magnetization-Temperature Curves for a 70 Per Cent Nickel-30 Per Cent Copper Powder Mixture.

close together, T_1 and T_2 . The magnetization drop, σ' , between these two temperatures is caused by the presence of a homogeneous alloy of which the Curie point lies between T_1 and T_2 . The specific magnetization of this alloy below the Curie point is given by

$$\sigma' = kmc$$

where k is a constant, m is the weight per cent of nickel at a given concentration, and c is the concentration. This equation follows, of course, from Marian's discovery that the magnetic moment of the

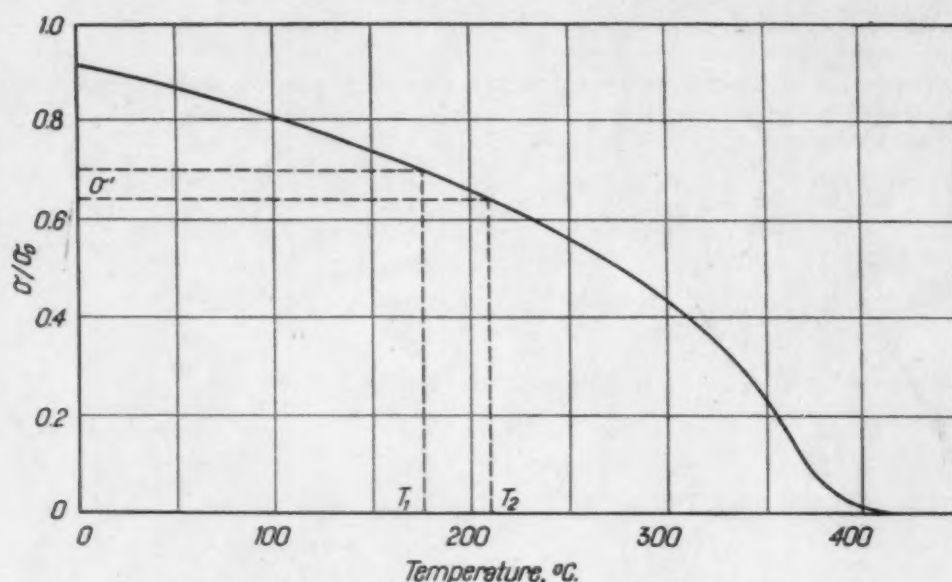


Fig. 4—Method for Interpreting Magnetization-Temperature Curves.

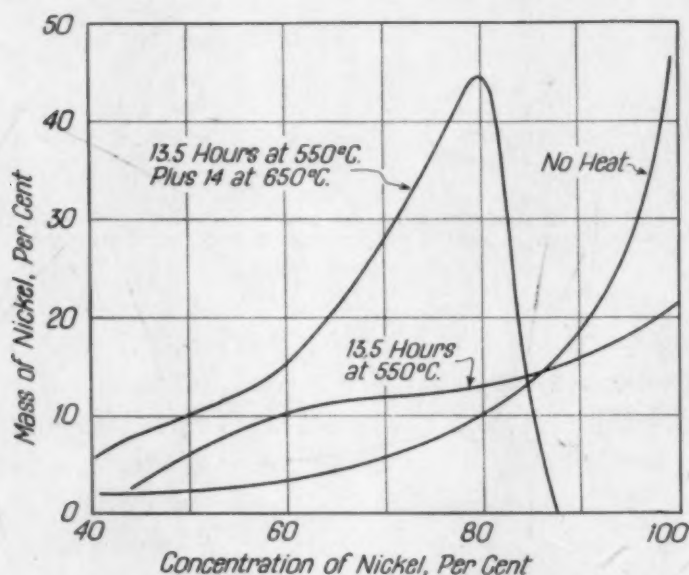


Fig. 5—Distribution of Nickel Concentrations in a 90 Per Cent Nickel-10 Per Cent Copper Powder Mixture at Several Stages.

nickel is a linear function of the concentration. This equation may be solved for m as follows: σ' is taken from the graph; k is determined from the curve for pure nickel; and c is the concentration for which the Curie temperature lies between T_1 and T_2 .

In this way tables may be constructed showing what weight of nickel is present at the various concentrations. Table I shows these data for a 90 per cent nickel-10 per cent copper alloy at three stages of sintering. These results are shown graphically in Fig. 5.

Table I
Distribution of Concentrations for 90 Per Cent Nickel-10 Per Cent Copper Alloy

No heat treatment				
T_1	T_2	Concentration Limits Per Cent	σ'^*	Weight Per Cent Nickel
0° C.	50° C.	43.5-51.1	0.5	4
50	100	51.1-59.0	0.5	4
100	150	59.0-67.0	1.0	6
150	200	67.0-75.0	1.5	8
200	250	75.0-82.7	2.0	10
250	300	82.7-90.7	3.2	14
300	350	90.7-98.0	6.3	26
350	400	98.0-100	10.0	38
Same after 13.5 hours heat treatment at 550 degrees Cent.				
T_1	T_2	Concentration Limits Per Cent	σ'	Weight Per Cent Nickel
0° C.	50° C.	43.5-51.1	0.6	5
50	100	51.1-59.0	1.3	9
100	150	59.0-67.0	2.0	12
150	200	67.0-75.0	2.1	11
200	250	75.0-82.7	2.6	13
250	300	82.7-90.7	3.3	14
300	350	90.7-98.0	4.7	19
350	400	98.0-100	5.4	20
Same after 13.5 hours at 550 degrees Cent. plus 14 hours at 650 degrees Cent.				
0° C.	50° C.	43.5-51.1	1.0	8
50	100	51.1-59.0	1.8	12
100	150	59.0-67.0	3.0	19
150	200	67.0-75.0	5.8	31
200	250	75.0-82.7	9.2	44
250	300	82.7-90.7	0.5	2
300	350	90.7-98.0	0	0
350	400	98.0-100	0	0

*In order to increase the accuracy, σ' is taken directly from the experimentally determined changes in weight on application of the magnetic field. The values shown are therefore larger than those shown in Fig. 2 where reduced magnetizations, σ/σ_0 , are plotted.

The data bring out a number of interesting facts. First, before sintering, the figures should indicate that 100 per cent of the nickel is undiluted. This is not the case, and must be attributed to a fault in the method. It arises from the fact that the Curie point is not a sharply defined temperature. The results show, however, that a very large proportion of the nickel is present as almost pure nickel. Second, after 13.5 hours heating at 550 degrees Cent. (1020 degrees Fahr.) the concentrations are well distributed between 40 and 100 per cent. There is still a substantial amount of pure nickel in the alloy, but there is a suggestion of a maximum at about 65 per cent. After long heating, homogeneity is approached and the distribution of concentration rises sharply at about 80 per cent. No doubt the peak would have approached 90 per cent if heating had been continued still longer. No pure nickel now remains, in fact the highest concentration of nickel is about 90 per cent.

These results are admittedly imperfect. The total mass percentages do not all add up to 100 per cent, and there are other dif-

ficulties. Nevertheless, it is believed that this will prove to be a useful tool in powder metallurgy. One of the most valuable results is that it is now possible to estimate the quantity of pure nickel remaining after sintering has been in progress for a certain time. Work is continuing on efforts to improve the apparatus and the analysis of results. The method is, of course, limited to the study of a ferromagnetic metal dissolving in a nonferromagnetic metal.

SUMMARY

A magnetic method, based on the analysis of magnetization-temperature curves, is developed and used to follow the approach to homogeneity in copper-nickel powder mixtures.

CHROMIUM STEELS OF LOW-CARBON CONTENT

BY RUSSELL FRANKS

Abstract

This paper describes the results of tests made on low-carbon steels containing up to about 25 per cent chromium. The investigation has shown that wrought 3 per cent chromium steels containing up to about 0.10 per cent carbon are at least five times as resistant to atmospheric corrosion in industrial atmospheres as ordinary low-carbon steel. The low-carbon 3 per cent chromium steel is relatively free of air hardening, as demonstrated by tests on samples air-cooled from 1650 degrees Fahr. After this treatment the steel is soft, ductile, and tough. Annealing at subcritical temperatures (1340 degrees Fahr.) also softens the steels, and imparts high ductility and toughness. The steels so heat treated have good toughness at temperatures down to -190 degrees Fahr. but their toughness is not so high at these low temperatures when the steel has been previously normalized at 1650 degrees Fahr. The low-carbon 3 per cent chromium steel can be welded; and the welds have good toughness at room temperature and at temperatures down to -40 degrees Fahr. The resistance of the low-carbon 3 per cent chromium steel to atmospheric deterioration makes it suitable for use in the form of articles such as roofing, gutters, drain pipes, and other parts exposed to weathering. Fence wire represents an article to be made from the steel, as well as certain structural parts for application in which atmospheric corrosion is a serious menace.

THIS investigation was carried out to determine the effects of chromium on such characteristics of low-carbon steel as resistance to atmospheric corrosion and oxidation at elevated temperatures, and susceptibility to air hardening. The steels for the investigation were melted in high frequency induction furnaces and were cast into ingots weighing up to approximately 50 pounds. In melting, only manganese and silicon were used as deoxidizers to obtain sound steel. The finished steels contained from 0.15 to 0.35 per cent

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silicon and from 0.30 to 0.60 per cent manganese. The phosphorus was limited to a maximum of 0.025 per cent and the sulphur to 0.035 per cent. Except in a few instances the percentage for carbon was limited to a maximum of 0.10 per cent. It was necessary to control all these constituents to this extent in order to determine the true effects of chromium. The small 2 to 3-inch square ingots representing the different steels were first forged to small billets or sheet bars, and subsequently rolled to $\frac{3}{4}$ or 1-inch rounds, and sheet or plate to obtain the samples for the various tests.

ATMOSPHERIC CORROSION TESTS

Twenty-gage sheet samples of the steels in the annealed and descaled conditions were used in the atmospheric corrosion tests. The samples were exposed to the industrial atmosphere in Long Island City, N. Y., for several years and subsequently transferred to Niagara Falls, N. Y., where they were exposed for a period of about eight years. In both instances the samples were exposed in the midst of industrial plants under conditions representative of those normally encountered in an industrial area. During the period of exposure the samples were cleaned twice, removing the loose oxide by means of a wire brush. They were cleaned once after an exposure period of about seven years and again after an exposure period which varied between ten and eleven years. For the sake of simplicity the results are expressed in terms of a 10-year period. As shown in Table I and in Fig. 1, the samples tested represented steel containing from zero to about 17 per cent chromium.

Table I
Results of Atmospheric Corrosion Tests on Low-Carbon Steels of Different Chromium Contents After 10-Year Exposure to an Industrial Atmosphere

Heat No.	Per Cent Chromium	Per Cent Carbon	Rate of Loss in Weight Grams Per Square Inch Per Year
Steel	0.15	0.11
B379	3.05	0.07	0.023
5 Per Cent Cr	5.60	0.07	0.021
B366	7.65	0.07	0.007
B317	9.65	0.12	0.0006
12 Cr	12.58	0.08	Nil
18 Cr	17.26	0.08	Nil

Samples of a low-carbon 2 per cent chromium steel were originally included in the tests but these were lost. The results show that with as little as 3 per cent chromium present, a low-carbon steel with

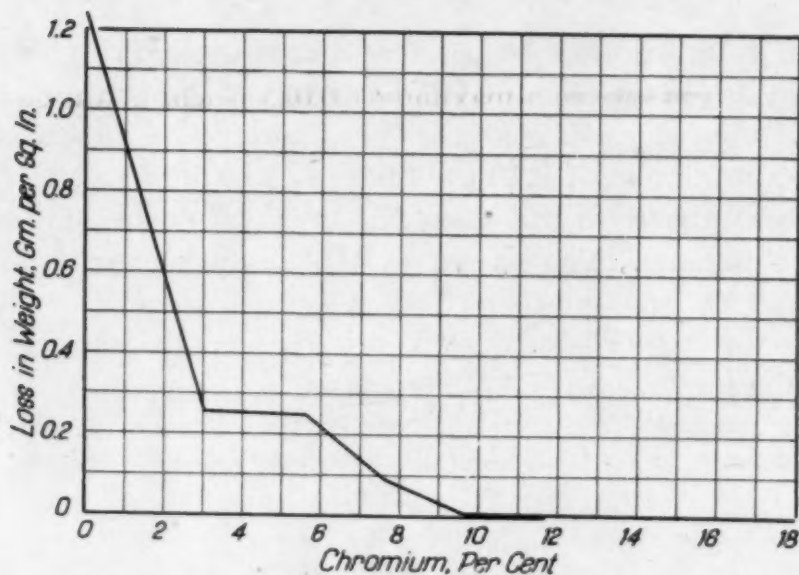


Fig. 1—Results of Atmospheric Corrosion Tests on Low-Carbon Steels of Different Chromium Contents After 10-Year Exposure to an Industrial Atmosphere.

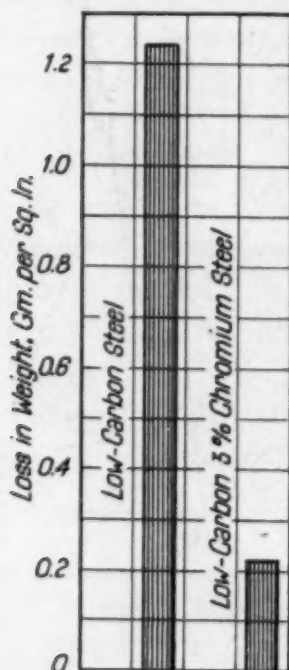
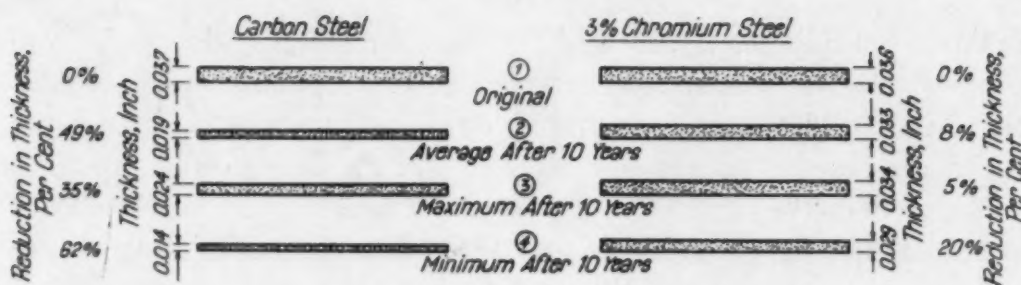


Fig. 2—Comparative Results of Atmospheric Corrosion Tests on Low-Carbon Steel and Low-Carbon 3.00 Per Cent Chromium Steel After a 10-Year Exposure to an Industrial Atmosphere.

excellent resistance to industrial atmospheres is obtained, and that as the chromium content is increased to 12 and 17 per cent the steels become practically immune to attack. As illustrated by Figs. 2 and



- ① Section Illustrating the Original Thickness of the Sheets.
- ② Section Illustrating the Average Thickness of the Sheets After a 10 Year Exposure to an Industrial Atmosphere.
- ③ Section Illustrating the Maximum Thickness in Certain Parts of the Sheets After the 10 Year Exposure.
- ④ Section Illustrating the Minimum Thickness in Certain Parts of the Sheets After the 10 Year Exposure.

Fig. 3—Comparative Results of Atmospheric Corrosion Tests on Low-Carbon Steel and on Low-Carbon 3.00 Per Cent Chromium Steel.

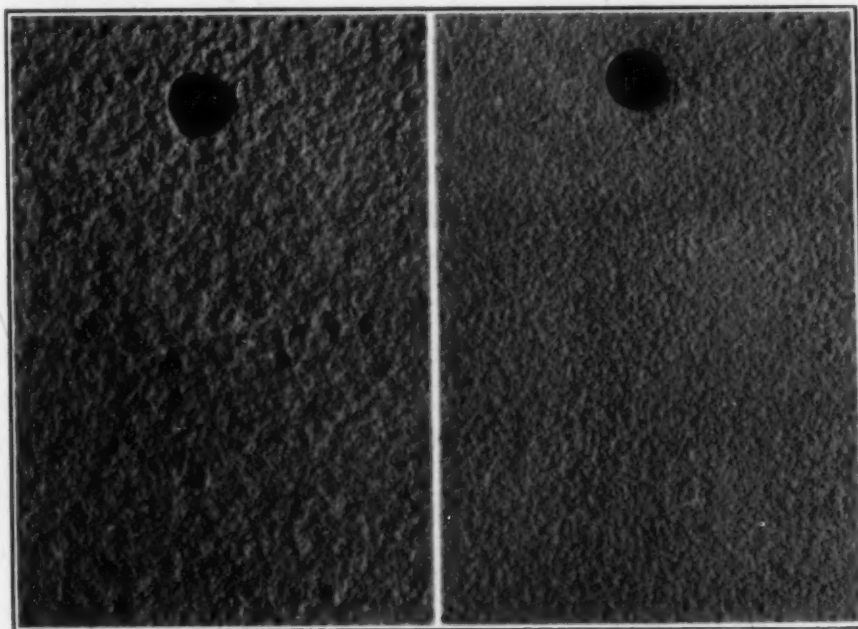


Fig. 4—Photographs Showing the Condition of the Surfaces of the Steels After the 10-Year Exposure Period to an Industrial Atmosphere. The weight loss for the carbon steel was 1.24 grams per square inch of surface, and for the low-carbon 3.00 per cent chromium steel was 0.26 gram per square inch of surface. $\times 1$. Left—Carbon steel. Right—3.00 per cent chromium steel (Heat B379).

3, the low-carbon 3 per cent chromium steel is approximately five times as resistant to atmospheric deterioration as ordinary carbon steel. Also, the data of Fig. 3 and 4 show that this degree of resistance to atmospheric deterioration is obtained in the absence of pitting. It is apparent that the low-carbon 3 per cent chromium steel corrodes similarly to ordinary carbon steel, with the exception that the oxide formed on the 3 per cent chromium steel does not readily

flake off but adheres tightly and prevents the metal underneath from wasting away so rapidly. The freedom from pitting in the atmosphere is especially well illustrated by Fig. 3, which compares the thicknesses of the carbon steel sample and the 3 per cent chromium steel sample before and after the 10-year exposure period. It should be stated, on the other hand, that the steels containing larger percentages of chromium extending up to about 9.5 per cent did exhibit a slight amount of pitting, although their weight losses were lower.

OXIDATION TESTS AT ELEVATED TEMPERATURES

The oxidation tests at elevated temperatures were made on 1½-inch long samples cut from 1-inch round bars and machined to a ⅞-inch diameter. The scale was removed from the samples prior to exposure to the high temperature by machining, to assist in obtaining uniform results. The samples were in the annealed condition and were exposed for the periods shown in Table II to an oxidizing atmosphere (air).

Table II
Results of Oxidation Tests on Low-Carbon Steels of Different Chromium Contents After 900 to 1000 Hours' Exposure at the Temperatures Shown

Heat No.	Per Cent Chromium	Per Cent Carbon	Per Cent Loss in Weight			
			1000 Hr. at 1000-1022 °F.	1000 Hr. at 1184-1202 °F.	900 Hr. at 1328-1346 °F.	1000 Hr. at 1454-1472 °F.
B527	0.09	0.37	8.49	26.50	58.80
B944	1.12	0.08	0.34	5.50
C205	2.57	0.07	3.79	19.80
B379	3.05	0.07	Nil	3.39	16.20
C105	3.67	0.06	Nil	2.90	11.30
B907	5.60	0.12	Nil	0.60	8.30	31.60
B909	7.68	0.09	0.20	4.64
B913	9.83	0.07	Nil
B236	13.17	0.12	Nil	2.76
A66	16.15	0.10	1.75
DA	23.61	0.20	0.30

The results of the oxidation tests are illustrated by the curves of Fig. 5. At about 1000 degrees Fahr. (540 degrees Cent.) the ordinary carbon steel exhibited fairly good resistance to oxidation, while the 3 per cent chromium steel underwent practically no loss in weight during the 1000-hour exposure period. At about 1200 degrees Fahr. (650 degrees Cent.) the carbon steel wasted away at a rapid rate as was the case with the steel containing about 1 per cent chromium, but the 3 per cent chromium steel exhibited substantial resistance to oxidation. With higher chromium contents the steels became practically free of scaling at this temperature. The losses

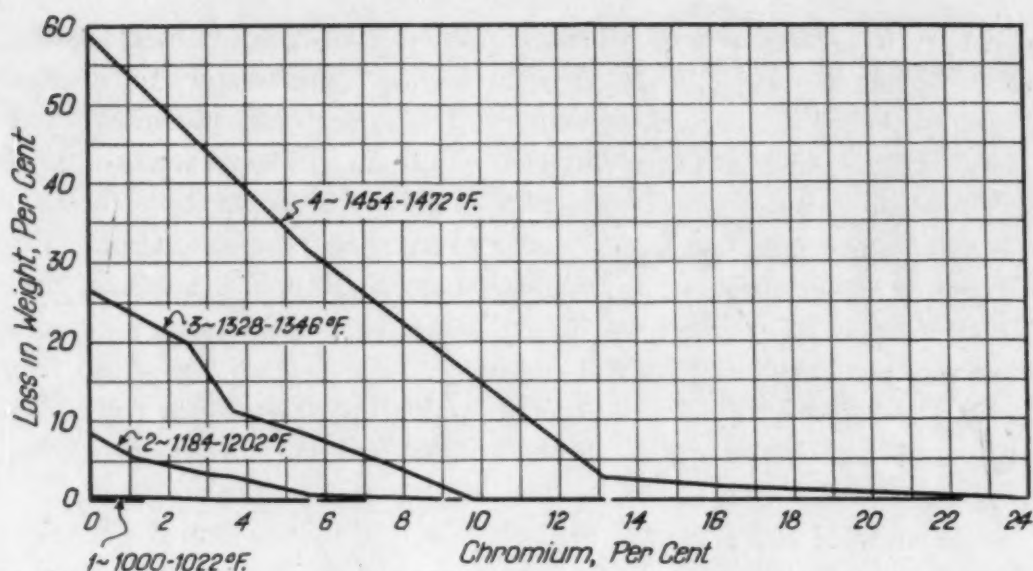


Fig. 5—Results of Oxidation Tests on Low-Carbon Steels of Different Chromium Contents After 900 to 1000 Hours' Exposure at Temperatures Shown.

for the ordinary carbon steel exposed in the range 1328-1346 degrees Fahr. (720-730 degrees Cent.) were exceedingly high, but decreased rapidly when the steel contained 3 and 3.5 per cent chromium, and reached a very low value when about 10 per cent chromium was present. At this point the steel became very resistant to scaling. In the curve illustrating these data there were two definite changes in regard to oxidation resistance; one occurring at about 3 per cent chromium and the other at about 10 per cent chromium. Although a change did occur with about 3 per cent chromium present, the losses for this steel were quite high. The data obtained at 1454-1472 degrees Fahr. (790-800 degrees Cent.) (Curve 4) showed that the steels containing below 13 per cent chromium underwent fairly high losses as a result of oxidation. At about 13 per cent chromium the oxidation losses were relatively low, and decreased further as the chromium content reached about 23 per cent. The steel containing this latter percentage of chromium remained substantially unaffected from the standpoint of oxidation during the 1000-hour test period.

SUSCEPTIBILITY TO AIR HARDENING

It is generally known that many of the low-carbon steels of low chromium contents are subject to air hardening¹. For example, when

¹E. C. Wright and P. F. Mumma, "Properties of Low-Carbon Medium-Chromium Steels of the Air-Hardening Type," T.P. No. 496, American Institute of Mining and Metallurgical Engineers, 1933.

ordinary low-carbon steel containing 5 per cent chromium is heated to 1650 degrees Fahr. (900 degrees Cent.) (above the Ac_1 point) and air-cooled, it is relatively hard and low in ductility, but when a low-carbon steel containing no chromium is similarly treated it is soft and quite ductile. The air hardening of the 5 per cent chromium steel has to be taken into consideration in fabrication, or difficulty will be encountered in welding, hot forming, hot bending, etc. Titanium and columbium additions² have been found to greatly reduce the air hardening of the 5 per cent chromium steels and are being used for this purpose. The reduction in air hardening is caused by a combination of the carbon with the titanium or columbium.

In view of the excellent resistance of the low-carbon 3 per cent chromium steel to atmospheric corrosion and oxidation at temperatures up to about 1200 degrees Fahr. (650 degrees Cent.) experiments were made to see whether this steel was as subject to air hardening as the 5 per cent chromium steel. In these experiments the chromium contents of the low-carbon steels ranged from 0 to slightly

Table III
Effect of Chromium on the Air Hardening of Low-Carbon Steel

Heat No.	Per Cent Cr	Per Cent C	Condition of Metal	Yield Str. at 0.20% Offset Psi.	Tensile Strength Psi.	% El. in 2 In.	% Red. of Area	Izod Impact Ft.-Lb.	Erichsen De-pression Mm.	Rockwell B Hardness	Brinell Hardness
J369	0.09	1	47,375	58,000	34	60	69	7.0	70	116
			2	38,330	57,000	33	72	68	8.4	68	107
			4	40,800	55,000	42	69	68	9.0	63	107
J356	2.08	0.098	1	52,500	68,250	37	73	72	7.0	78	137
			2	47,000	60,500	40	76	74	8.5	72	121
			4	37,000	67,500	36	69	68	8.2	79	137
C279	2.60	0.06	3	46,500	60,000	42	79	85	121
			4	38,500	68,000	35	72	89	146
J309	3.03	0.06	1	61,500	81,000	23	59	85	8.0	85	163
			2	52,000	61,500	35	77	92	9.5	66	128
			4	26,000	65,000	35	65	80	9.0	75	131
J367	2.98	0.071	1	57,000	80,500	22	51	67	7.4	82	153
			2	50,000	63,000	35	77	74	8.8	75	126
			4	28,000	70,000	32	59	57	8.5	78	149
C105	3.67	0.06	3	43,500	65,000	37	78	100	131
			4	56,500	91,000	20	50	38	286
J355	4.53	0.073	1	71,000	124,000	12	31	43	6.4	103	255
			2	44,000	71,750	35	77	81	9.0	83	153
			4	89,200	143,500	12	35	18	5.9	107	311
D267	6.08	0.08	1	149,000	181,000	5	12	22	5.5	..	340
			3	54,000	83,500	31	74	91	153
			4	163,000	197,000	6	20	19	351

1 = As-hot-rolled.

2 = Heated 2 hr. at 1340 degrees Fahr. and air-cooled.

3 = Heated 2 hr. at 1380 degrees Fahr. and air-cooled.

4 = Heated 10 min. at 1650 degrees Fahr. and air-cooled.

²F. M. Becket and R. Franks, "Titanium and Columbium in Plain High-Chromium Steels," *Transactions, American Institute of Mining and Metallurgical Engineers*, Vol. 113, 1934, p. 126-142.

over 6 per cent. The steels were tested in the as-hot-worked condition, after softening by use of a subcritical annealing treatment, and after air cooling from 1650 degrees Fahr. (900 degrees Cent.) (above the A_{c1} critical point). The samples of the steels were taken from 1-inch round bars and were tested for strength, ductility, toughness, and hardness; the data are given in Table III and show that low-carbon steels containing up to and slightly over 3 per cent chromium were soft, ductile, and tough after air cooling from 1650 degrees Fahr. (900 degrees Cent.), indicating relative freedom from air hardening. These steels were also soft, ductile, and tough after they were given the subcritical annealing treatment at 1340 degrees Fahr. (725 degrees Cent.). When the chromium content reached 3.6 per cent, even though the carbon content was kept low, the steel exhibited

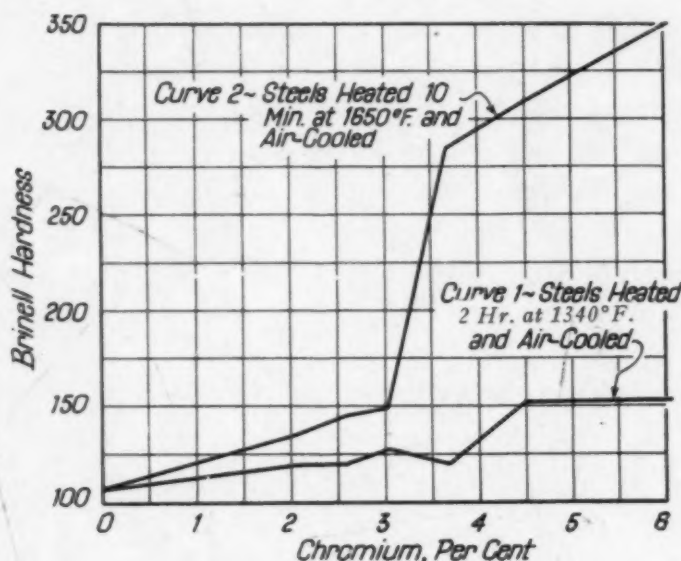


Fig. 6—Effect of Chromium on the Air Hardening of Low-Carbon Steel.

relatively strong air hardening characteristics on air cooling from 1650 degrees Fahr. (900 degrees Cent.). As the chromium content was increased respectively to 4.5 and 6 per cent, the susceptibility to air hardening increased but at a much slower rate than that which took place on raising the chromium content from slightly over 3 to 3.6 per cent.

It was concluded from these results, which are graphically illustrated in Fig. 6, that so long as the chromium content of the low-carbon steel was at a maximum of about 3.25 per cent, it would be substantially free of air hardening. It should be mentioned that the

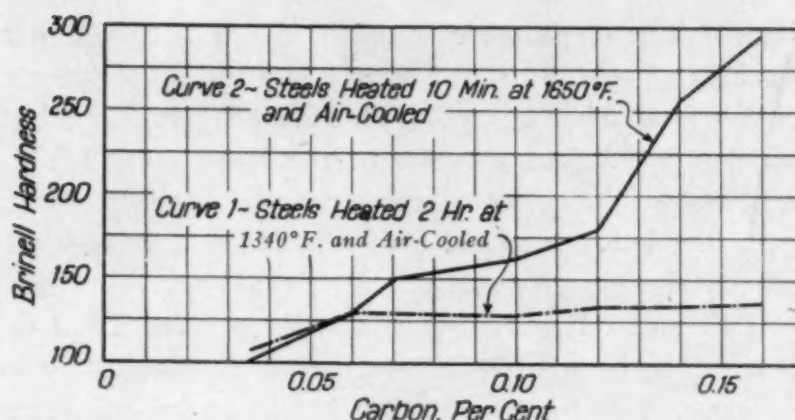


Fig. 7—Effect of Carbon on the Air Hardening of 3.00 Per Cent Chromium Steel.

low-carbon 3 per cent chromium steel given the subcritical annealing treatment at 1340 degrees Fahr. (725 degrees Cent.) had a high yield strength with maximum ductility and toughness.

It was recognized that carbon as well as chromium had a marked influence on air hardening, and in another series of experiments the effect of carbon on the 3 per cent chromium steel was studied. In these tests the chromium content was held at about 3 per cent, and the carbon content was increased to 0.16 per cent. Samples taken

Table IV
Effect of Carbon on the Air Hardening of 3 Per Cent Chromium Steel

Heat No.	Per Cent Cr	Per Cent C	Condition of Metal	Yield Str. at 0.20% Offset Psi.	Tensile Strength Psi.	% El. in 2 In.	% Red. of Area	Izod Impact Ft.-Lb.	Erichsen Mm. of De-pression	Rockwell B Hardness	Brinell Hardness
J365	3.07	0.035	1	30,750	54,500	35	66	70	6.7	68	116
			2	42,500	51,500	43	82	70	9.6	64	107
			3	18,750	52,000	43	77	68	9.5	58	101
J309	3.03	0.06	1	61,500	81,000	23	59	85	8.0	85	163
			2	52,000	61,500	35	77	92	9.5	66	128
			3	26,000	65,000	35	65	80	9.0	75	131
J367	2.98	0.071	1	57,000	80,500	22	51	67	7.4	82	153
			2	50,000	63,000	35	77	74	8.8	75	126
			3	28,000	70,000	32	59	57	8.5	78	149
J310	3.13	0.10	1	56,750	90,000	21	36	48	7.0	85	179
			2	52,500	63,750	34	74	86	9.0	72	128
			3	33,000	80,000	25	47	50	8.0	88	163
J366	3.07	0.12	1	46,000	101,500	16	27	28	5.4	87	156
			2	45,000	65,500	35	75	77	8.2	74	134
			3	40,000	87,000	22	40	32	6.6	90	179
J368	3.07	0.14	1	54,250	92,750	17	47	28	4.5	88	179
			2	48,500	70,000	34	76	77	8.4	76	134
			3	52,000	98,000	20	40	30	5.7	100	255
J311	3.15	0.16	1	66,000	125,750	11	16	15	5.9	104	228
			2	54,000	72,500	33	75	91	8.0	82	137
			3	73,000	138,000	12	22	14	5.6	106	293

1 = As-hot-rolled.

2 = Heated 2 hr. at 1340 degrees Fahr. and air-cooled.

3 = Heated 10 min. at 1650 degrees Fahr. and air-cooled.

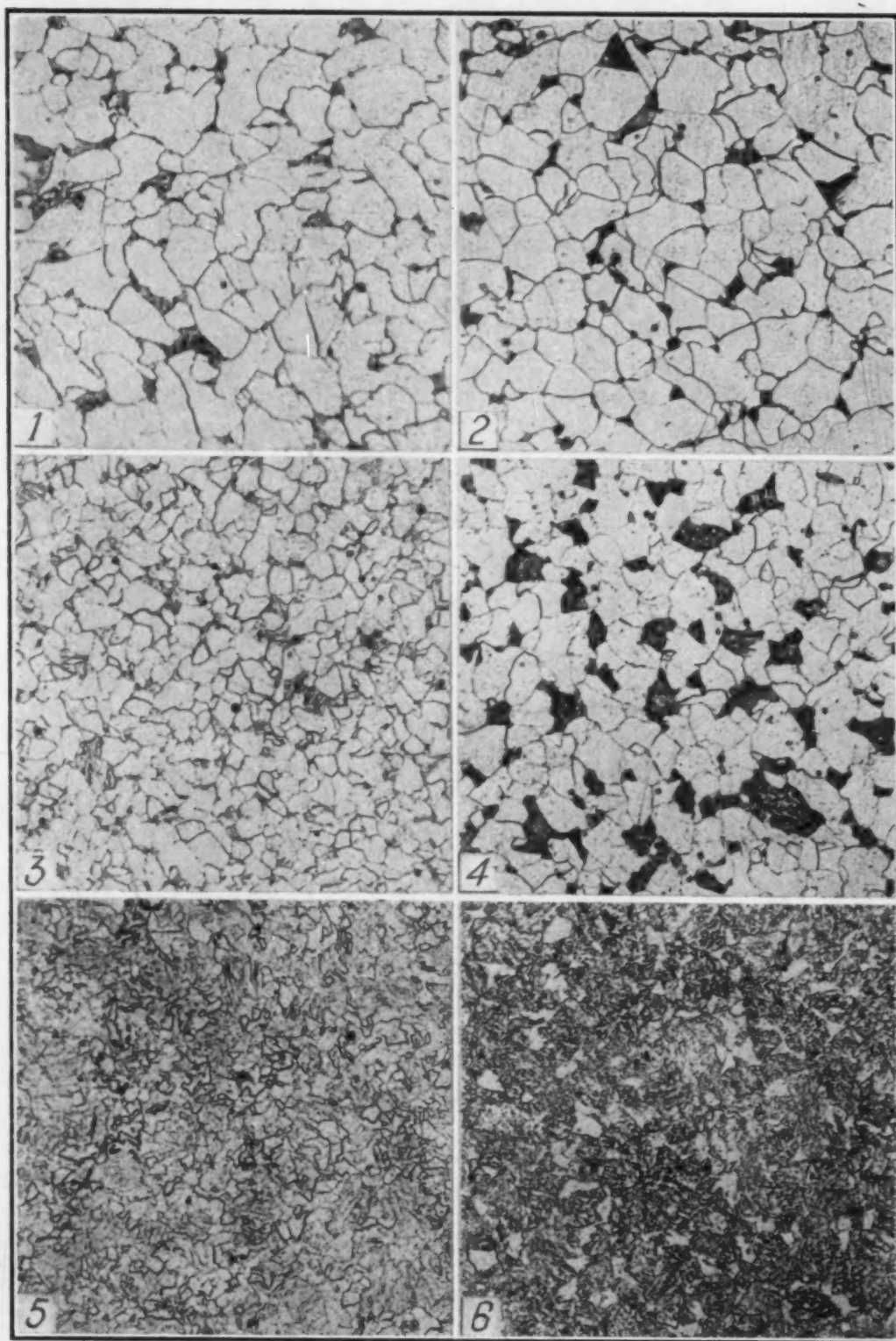


Fig. 8—See Caption on Next Page.

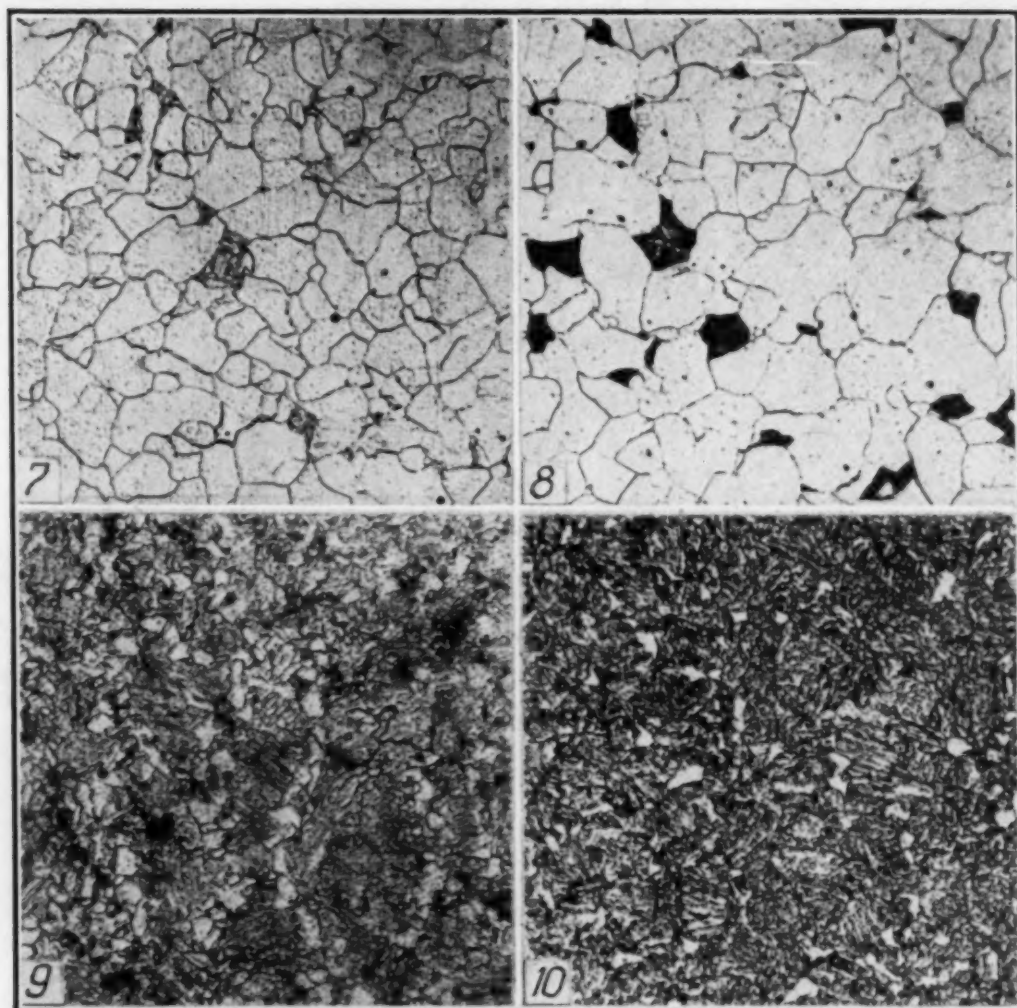


Fig. 8—Photomicrographs of Structures of Steels After Treatments 1 and 2. $\times 250$. Odd Numbers Treatment 1—Heated 2 Hours at 1340 Degrees Fahr. and Air-Cooled. Even Numbers Treatment 2—Heated 10 Minutes at 1650 Degrees Fahr. and Air-Cooled. Nos. 1 and 2—0.09 per cent carbon steel. Nos. 3 and 4—3.00 per cent chromium—0.06 per cent carbon steel. Nos. 5 and 6—4.5 per cent chromium—0.07 per cent carbon steel. Nos. 7 and 8—3.00 per cent chromium—0.035 per cent carbon steel. Nos. 9 and 10—3.00 per cent chromium—0.16 per cent carbon steel.

from bars of the steels were tested in like manner after annealing at subcritical temperatures and after normalizing at 1650 degrees Fahr. (900 degrees Cent.). The data are described in Table IV and Fig. 7.

It will be noted that the carbon content of the steels was progressively increased from a minimum of 0.035 per cent to a maximum of 0.16 per cent, using relatively small increments. As the carbon content was increased to 0.12 per cent there was a steady but relatively small increase in the hardness of the steels on air cooling from the normalizing temperature (1650 degrees Fahr.), but when the carbon content was increased to 0.14 and 0.16 per cent, the hard-

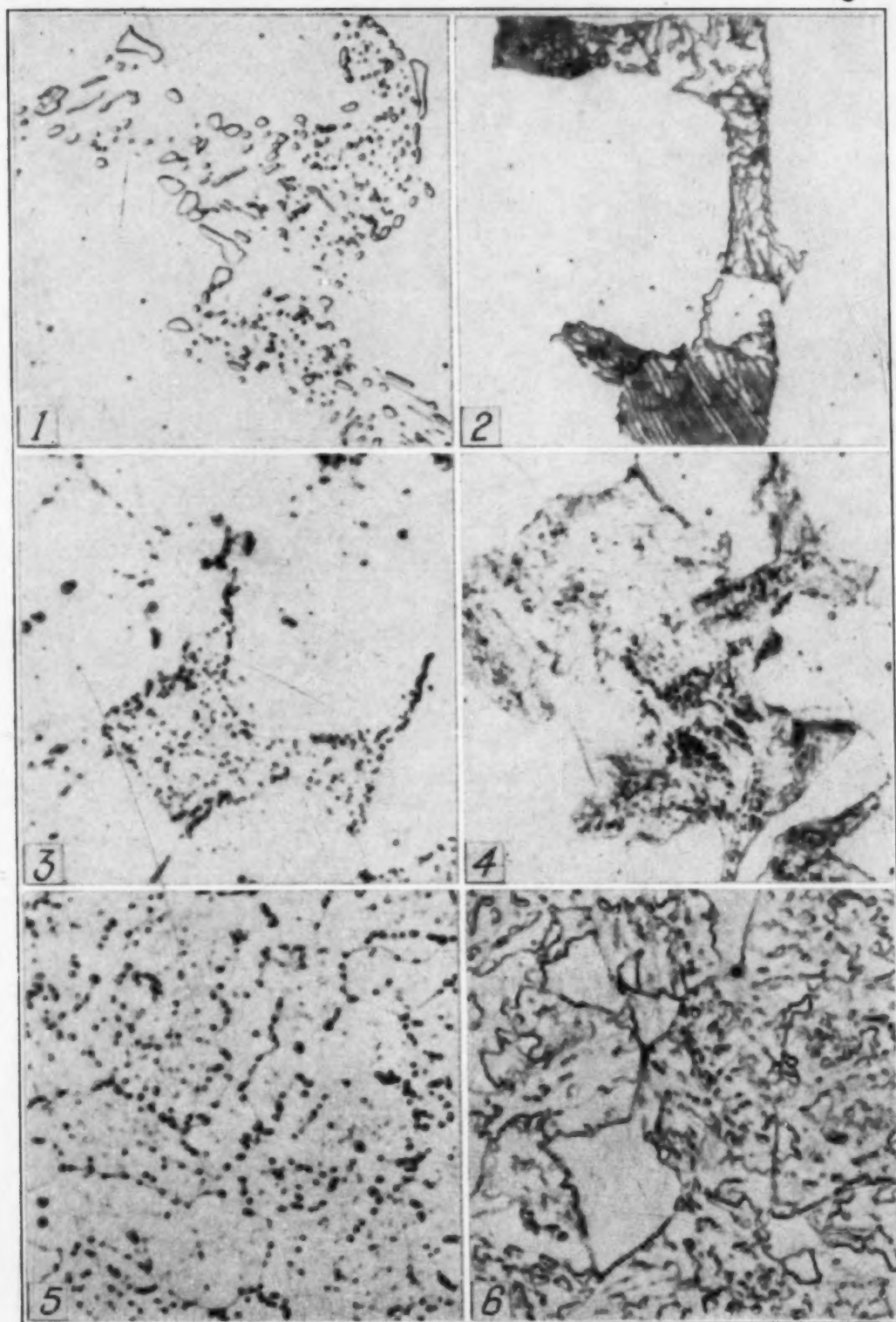


Fig. 9—See Caption on Next Page.

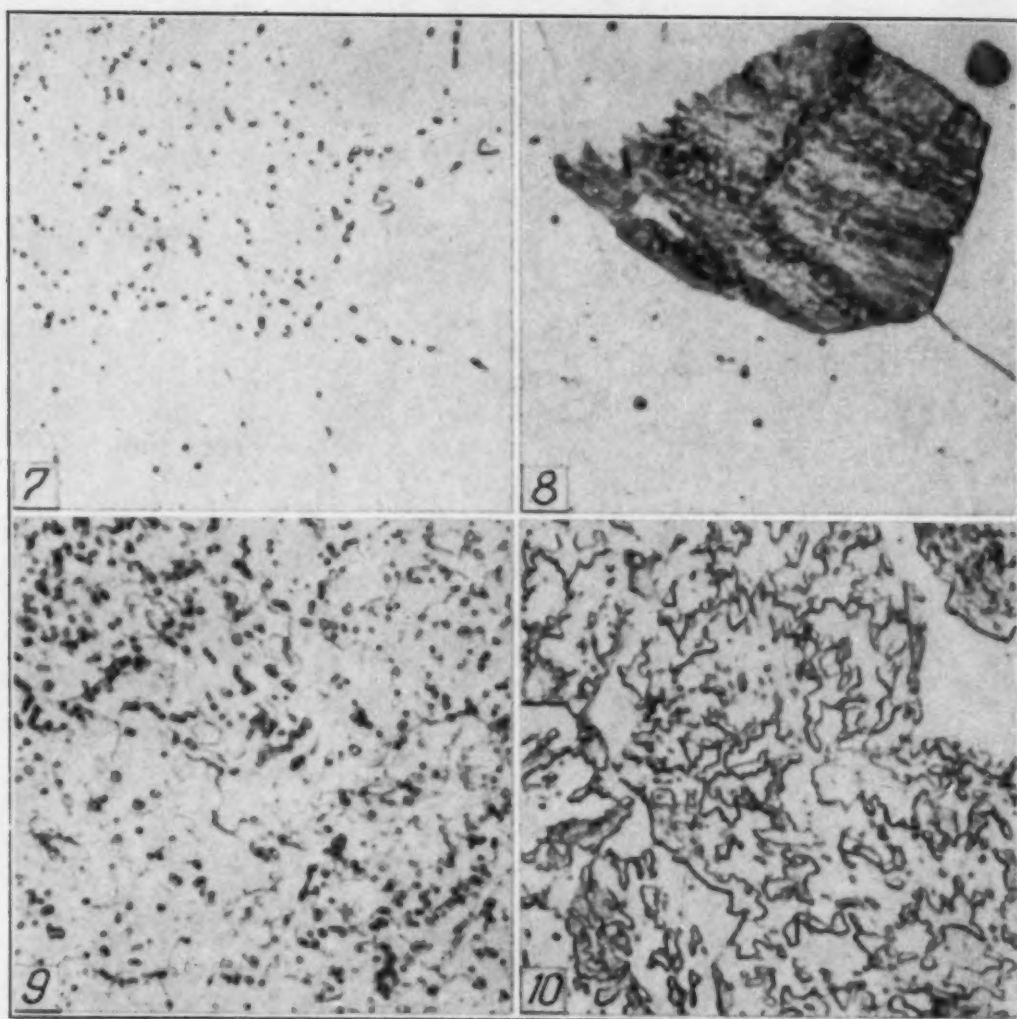


Fig. 9—Photomicrographs of Structures of Steels After Treatments 1 and 2. $\times 2000$. Odd Numbers Treatment 1—Heated 2 Hours at 1340 Degrees Fahr. and Air-Cooled. Even Numbers Treatment 2—Heated 10 Minutes at 1650 Degrees Fahr. and Air-Cooled. Nos. 1 and 2—0.09 per cent carbon steel. Nos. 3 and 4—3.00 per cent chromium—0.06 per cent carbon steel. Nos. 5 and 6—4.5 per cent chromium—0.07 per cent carbon steel. Nos. 7 and 8—3.00 per cent chromium—0.035 per cent carbon steel. Nos. 9 and 10—3.00 per cent chromium—0.16 per cent carbon steel.

ness of the steels increased to a considerable extent. In fact, the steel containing 0.12 per cent carbon was harder and less ductile and tough than the steels containing up to 0.10 per cent carbon after the treatment at 1650 degrees Fahr. (900 degrees Cent.). The results of the Erichsen tests were particularly indicative, and it was concluded that to avoid difficulties due to air hardening, the carbon content of the 3 per cent chromium steel should be at a maximum of 0.10 per cent and preferably no higher than 0.08 per cent.

The microstructures of the steels included in this part of the investigation were studied to determine why the low-carbon 3 per

cent chromium steel was relatively free of air hardening, whereas the low-carbon 5 per cent chromium steel was quite susceptible to air hardening; and also why increasing the carbon content of the 3 per cent chromium steel made this steel quite subject to air hardening.

The structures of the steels are illustrated by the photomicrographs of Figs. 8 and 9. Fig. 8 shows the structures of the steels at a magnification of 250 diameters, and Fig. 9 at a magnification of 2000 diameters. These photomicrographs reveal that the structure of the ordinary low-carbon steel consists of ferrite and a small amount of pearlite, after air cooling from 1650 degrees Fahr. (900 degrees Cent.), and ferrite and spheroidized carbides after heating at 1340 degrees Fahr. (725 degrees Cent.) and air cooling. It is difficult to interpret the structure obtained on air cooling the low-carbon 3 per cent chromium steel from 1650 degrees Fahr. (900 degrees Cent.). After this heat treatment, the structure of the steel consists of ferrite containing a decomposition product of austenite, which is unlike pearlite and does not appear to be pseudomartensite. This type of structure is observed in the 3 per cent chromium steels containing up to about 0.10 per cent carbon. The constituent represents a small part of the structure and is contained in soft ferrite, explaining why the low-carbon 3 per cent chromium steel like ordinary low-carbon steel is soft after air cooling from above the A_{c1} point. After air cooling from 1340 degrees Fahr. (725 degrees Cent.), the carbides present in the low-carbon 3 per cent chromium steel have spheroidized in much the same manner as the carbides in the ordinary carbon steel when similarly heat treated.

On the other hand, after air cooling the low-carbon 4.50 per cent chromium steel from 1650 degrees Fahr. (900 degrees Cent.), its structure consists primarily of a pseudomartensitic type of structure containing a small amount of chromium ferrite, which explains why this steel hardens on air cooling from above the A_{c1} point. As was to be expected, heating the 4.50 per cent chromium steel at 1340 degrees Fahr. (725 degrees Cent.) causes the carbides to spheroidize, leaving a chromium ferrite matrix containing carbides. The steel in this condition is soft and ductile. When heated above the A_{c1} point, it is evident that the carbides of the higher chromium steel act quite unlike the carbides of the ordinary low-carbon steel or of the low-carbon 3 per cent chromium steel. The carbides of the low-carbon 4.50 per cent chromium steel are probably rich in chromium,

which would account for their greater influence on air hardening.

The last four photomicrographs of Figs. 8 and 9 show the effect of carbon on the structure of the 3 per cent chromium steel. It is clear that when the carbon content of this steel is increased from 0.035 to 0.16 per cent its structure on air cooling from 1650 degrees Fahr. (900 degrees Cent.) consists of a pseudomartensitic type of constituent containing some ferrite. The presence of so much of the pseudomartensitic type of constituent in the 3 per cent chromium steel containing 0.16 per cent carbon causes the metal to air harden in much the same way as when the carbon content is kept low and the chromium content is increased. Thus to avoid serious air hardening it is necessary to limit both the chromium and carbon contents.

TESTS AT SUBNORMAL TEMPERATURES ON LOW-CARBON 3 PER CENT CHROMIUM STEELS

There are many applications in which retention of toughness at low temperatures is an important factor. The low-carbon 3 per cent chromium steels were subjected to temperatures as low as -300 degrees Fahr. (-185 degrees Cent.) to see how their toughness would be affected. The steel treated at these temperatures was given the subcritical annealing treatment at 1340 degrees Fahr. (725 degrees Cent.), and the normalizing treatment at 1650 degrees Fahr. (900 degrees Cent.). Samples of the steel were also given the normalizing heat treatment followed by reheating at the subcritical temperature, and the results are presented in Table V.

These tests showed that after annealing at 1340 degrees Fahr. (725 degrees Cent.) the steel retained its toughness to a marked degree at temperatures down to -190 degrees Fahr. (-125 degrees Cent.). The steel in this condition of heat treatment had substantially the same degree of toughness at these low temperatures as it had at room temperature, but when the temperature was decreased to -300 degrees Fahr. (-185 degrees Cent.) it became embrittled. The results of the tests on the normalized low-carbon 3 per cent chromium steel samples were different. They showed that the steel underwent a marked decrease in toughness as the temperature dropped below -90 degrees Fahr. (-68 degrees Cent.). It should be pointed out however, that at -150 degrees Fahr. (-100 degrees Cent.) the steel had 20 ft-lb. of impact, which was considered substantial. In general these results revealed that the steel was not

in as stable a condition after normalizing as it was after softening at 1340 degrees Fahr. (725 degrees Cent.). This was substantiated by the fact that the samples given the normalizing heat treatment, followed by heating at 1380 degrees Fahr. (750 degrees Cent.) re-

Table V
Results of Impact Tests at Low Temperature on Low-Carbon 3 Per Cent Chromium Steels

Composition: Chromium3.07 Per Cent		
Manganese0.40 Per Cent		
Silicon0.25 Per Cent		
Carbon0.075 Per Cent		
Heat Treatment	Temperature of Test	Izod Impact Ft.-Lb.
1	Room temperature	86
2	Room temperature	77
3	Room temperature	56
1	32 Degrees Fahr.	82
2	32 Degrees Fahr.	78
3	32 Degrees Fahr.	56
1	-40 Degrees Fahr. (-40 Degrees Cent.)	83
2	-40 Degrees Fahr. (-40 Degrees Cent.)	78
3	-40 Degrees Fahr. (-40 Degrees Cent.)	41
1	-90 Degrees Fahr. (-68 Degrees Cent.)	81
2	-90 Degrees Fahr. (-68 Degrees Cent.)	90
3	-90 Degrees Fahr. (-68 Degrees Cent.)	35
1	-150 Degrees Fahr. (-100 Degrees Cent.)	83
2	-150 Degrees Fahr. (-100 Degrees Cent.)	92
3	-150 Degrees Fahr. (-100 Degrees Cent.)	20
1	-190 Degrees Fahr. (-125 Degrees Cent.)	76
2	-190 Degrees Fahr. (-125 Degrees Cent.)	80
3	-190 Degrees Fahr. (-125 Degrees Cent.)	18
1	-300 Degrees Fahr. (-185 Degrees Cent.)	4
2	-300 Degrees Fahr. (-185 Degrees Cent.)	2
3	-300 Degrees Fahr. (-185 Degrees Cent.)	3

1 = Heated 2 hr. at 1340 degrees Fahr. and air-cooled.

2 = Heated 10 min. at 1650 degrees Fahr. and air-cooled. Reheated 3 hr. at 1380 degrees Fahr. and air-cooled.

3 = Heated 10 min. at 1650 degrees Fahr. and air-cooled.

tained their toughness equally as well at temperatures down to -190 degrees Fahr. (-125 degrees Cent.) as those given only the annealing treatment at 1340 degrees Fahr. (725 degrees Cent.). Thus, the condition of the carbide in the low-carbon 3 per cent chromium steels had an important influence on their low temperature toughness.

WELDING LOW-CARBON 3 PER CENT CHROMIUM STEEL

The welding properties of the low-carbon 3 per cent chromium steel were considered to be especially important in view of its low air hardening tendencies, so tests were made on plates of different thicknesses, joined together by arc welding. The welding rod employed was similar in composition to that of the plate metal. The welds were tested for strength, ductility, and toughness, and the results are given on the next page in Table VI.

These tests were made on all-weld-metal $\frac{3}{8}$ -inch diameter samples prepared from welds made in $\frac{9}{16}$ -inch thick low-carbon 3 per cent chromium steel plate. The plate was previously softened by heating 2 hours at 1340 degrees Fahr. (725 degrees Cent.) and air cooling. The results showed that while the weld metal was ductile and tough in the as-welded condition, both of these properties were improved by stress relieving at 1200 degrees Fahr. (650 degrees Cent.), and annealing at 1340 degrees Fahr. (725 degrees Cent.). The condition of this weld and the adjacent areas in regard to hardness is shown by the data of Fig. 10.

This weld was made in plate containing 3.01 per cent chromium and 0.07 per cent carbon. The data show that the weld is fairly soft in the as-welded condition, and that its hardness is reduced on stress relieving at 1200 degrees Fahr. (650 degrees Cent.). The weld, however, is harder in both conditions than the plate metal, the hardness of which is not materially increased in the areas adjacent to the weld. This explains why the weld is ductile and tough.

An additional arc weld was made in the annealed $\frac{9}{16}$ -inch thick 3 per cent chromium steel plate with 3 per cent chromium steel

Table VI
Mechanical Properties of Weld Metal Containing 2.96 Per Cent Chromium
and 0.08 Per Cent Carbon

Condition of Sample	Yield* Strength Psi.	Tensile Strength Psi.	Per Cent Elonga- tion in 1½ In.	Per Cent Reduction of Area	Izod Impact Ft.-Lb.
As-welded	47,200	75,400	19	43	33
Stress relieved 2 hr. at 1200 degrees Fahr., and air-cooled	48,100	74,500	24	68	47
Heated 2 hr. at 1340 degrees Fahr. and air-cooled	48,000	74,500	25	74	43

*Determined with dividers.

rods. The weld contained 2.78 per cent chromium and 0.06 per cent carbon. The weld was tested for toughness both at room temperature and at subnormal temperatures, and the results are shown in Table VII. See page 634.

The impact tests showed that in the as-welded state, the weld had an Izod impact value of 28 ft-lb. at room temperature, which was decreased to about 20 ft-lb. on cooling to 32 degrees Fahr. The weld exhibited similar toughness on cooling to -40 degrees Fahr., whereas on cooling to -90 degrees Fahr., a marked drop in toughness occurred. The weld stress-relieved at 1200 degrees Fahr. had

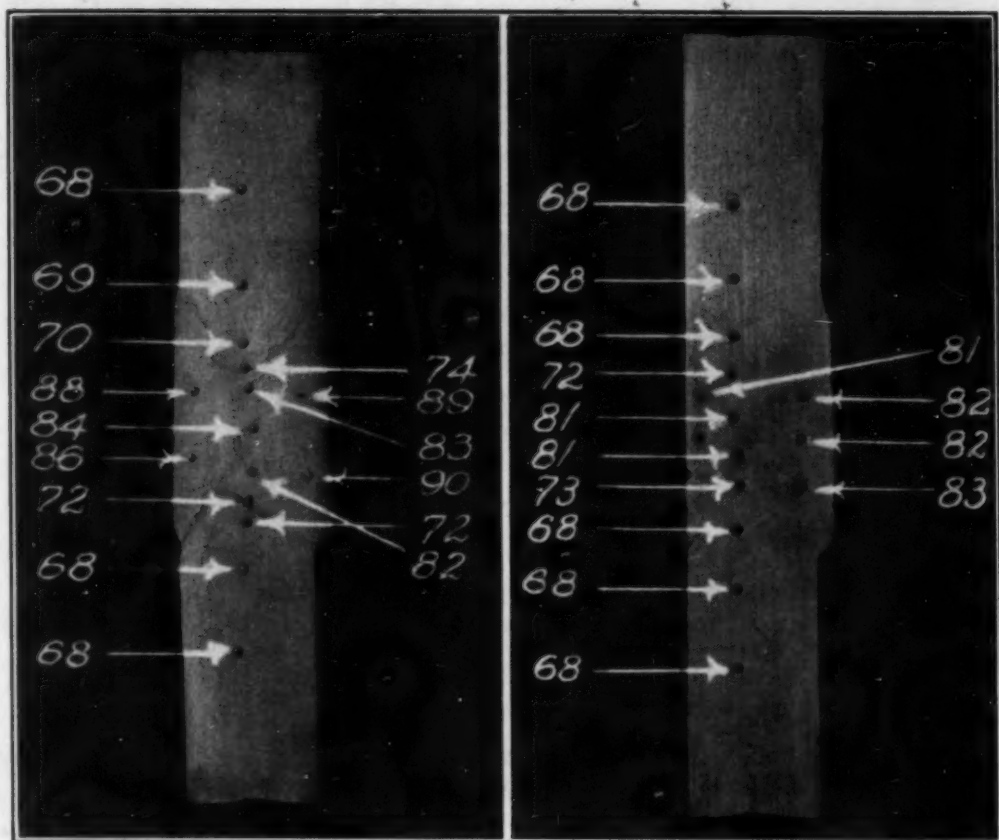


Fig. 10—Rockwell "B" Hardness Values on Arc-Welded Samples of Low-Carbon 3.00 Per Cent Chromium Steel. $\times 1$. The photograph on the left represents a sample in the as-welded condition, while the photograph on the right represents a sample stress-relieved 2 hours at 1200 degrees Fahr. and air-cooled.

higher toughness at room temperature, but decreased in toughness to the same extent on cooling to the lower temperatures as it did in the as-welded condition. The results on the weld samples held 2 hours at 1340 degrees Fahr. (725 degrees Cent.) and air-cooled showed that this treatment produced an improvement in the toughness of the weld at room temperature, and at temperatures down to -40 degrees Fahr. (-40 degrees Cent.). The weld heat treated at 1340 degrees Fahr. (725 degrees Cent.) also exhibited good toughness at -90 degrees Fahr. (-68 degrees Cent.), but at -150 degrees Fahr. (-100 degrees Cent.) was embrittled.

Tests were next made on arc welds produced in $\frac{1}{4}$ -inch thick plate of the same composition. This plate was also softened prior to welding by heating 2 hours at 1340 degrees Fahr. (725 degrees Cent.), and air cooling. The welds were prepared with rods of the same composition as those used in welding the $\frac{9}{16}$ -inch thick plate

Table VII
Impact Tests on 3 Per Cent Chromium Steel Weld

Condition of Sample	Izod Impact of Weld in Ft.-Lb.	Temperature of Test
As-welded	28	Room Temp.
As-welded	21	32° F.
As-welded	20	-40° F.
As-welded	6	-90° F.
Stress-relieved 2 hr. at 1200 degrees Fahr., and air-cooled	43	Room Temp.
Stress-relieved 2 hr. at 1200 degrees Fahr., and air-cooled	21	32° F.
Stress-relieved 2 hr. at 1200 degrees Fahr., and air-cooled	20	-40° F.
Stress-relieved 2 hr. at 1200 degrees Fahr., and air-cooled	6	-90° F.
Heated 2 hr. at 1340 degrees Fahr., and air-cooled.....	40	Room Temp.
Heated 2 hr. at 1340 degrees Fahr., and air-cooled.....	31	32° F.
Heated 2 hr. at 1340 degrees Fahr., and air-cooled.....	30	-40° F.
Heated 2 hr. at 1340 degrees Fahr., and air-cooled.....	10	-90° F.
Heated 2 hr. at 1340 degrees Fahr., and air-cooled.....	4	-150° F.

for the low temperature impact tests, and the results are given in Table VIII.

As shown by the data of the table, these welds were subjected to tensile and free bend tests. The tensile tests revealed that in the as-welded state the coupon sample fractured outside the weld, showing that the weld was stronger than the plate. The same results were obtained after stress relieving the welded sample at 1200 degrees Fahr. However, in both instances the sample exhibited good ductility before fracture that occurred in the plate away from the area adjacent to the weld. The fact that the welds and the metal adjacent to the welds had good ductility was also brought out in the free bend tests. In these tests the elongation in $\frac{1}{2}$ inch across the welds was 18 per cent as-welded, and 30 per cent on stress relieving at 1200 degrees Fahr. (650 degrees Cent.), and on fully softening with the subcritical annealing treatment at 1340 degrees Fahr. (725 de-

Table VIII
Tensile and Bend Tests on Arc Welds in $\frac{1}{4}$ -Inch Thick Plate Weld Containing 2.78 Per Cent Chromium and 0.06 Per Cent Carbon

Condition of Sample	Results of Tensile Tests			Results of Free Bend Test	
	Yield* Strength Psi.	Tensile Strength Psi.	Per Cent Elongation in 2 In.	Angle of Bend	Per Cent Elongation in $\frac{1}{2}$ In.
As-welded	40,800	65,200	17	Cracked on bending 180°	18
Welded sample stress-relieved 2 hr. at 1200° F. and air- cooled	35,300	66,600	34	180° without cracking	30
Welded sample heated 2 hr. at 1340° F. and air-cooled	180° without cracking	30

Note: Tensile samples broke outside weld.
 *Determined with dividers.

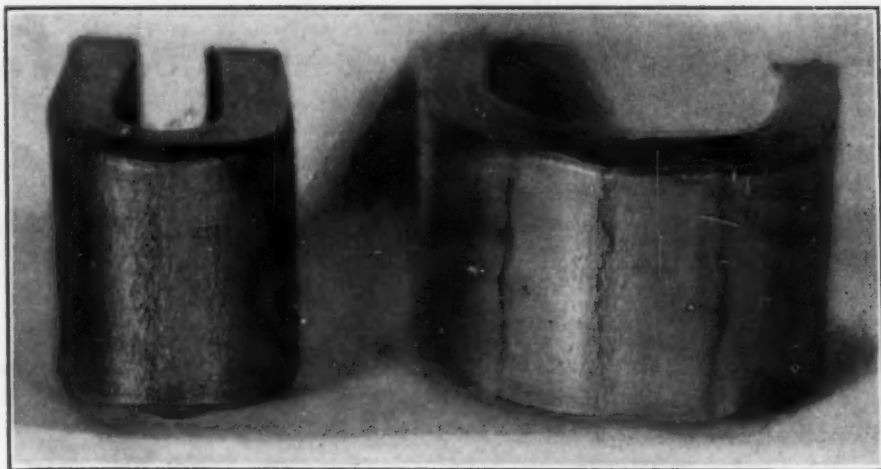


Fig. 11—Photograph Illustrating the Condition of the Weld and Adjacent Areas After the Free Bend Tests on the Welded 3.00 Per Cent Chromium Steel Samples. $\times 1$.

grees Cent.). The appearance of the samples subjected to the free bend tests are shown in Fig. 11.

The picture on the right shows the condition of the as-welded sample, while the picture on the left shows the condition of the stress-relieved sample after bending. The absence of brittleness both in the weld and adjacent areas is well illustrated by this photograph. The as-welded sample cracked in the weld on bending, but as previously stated, this did not occur until the weld metal had elongated 18 per cent.

SUMMARY

This investigation has shown that wrought 3 per cent chromium steel containing up to 0.10 per cent carbon is at least five times as resistant to atmospheric corrosion in industrial atmospheres as ordinary low-carbon steel. At elevated temperatures the low-carbon 3 per cent chromium steel is superior to ordinary carbon steel. It possesses good resistance to oxidation at temperatures up to about 1200 degrees Fahr. (650 degrees Cent.), whereas ordinary carbon steel is considered unsuitable for temperatures in excess of about 1000 degrees Fahr. (540 degrees Cent.).

The low-carbon 3 per cent chromium steel is relatively free of air hardening, as demonstrated by tests on samples air-cooled from 1650 degrees Fahr. (900 degrees Cent.). After this treatment the steel is soft, ductile, and tough. Annealing at subcritical tempera-

tures (1340 degrees Fahr.) also softens the steel and imparts high ductility and toughness with a comparatively high yield strength at 0.2 per cent offset. The steels have good toughness at temperatures down to -190 degrees Fahr. (-125 degrees Cent.) when previously given the subcritical annealing treatment at 1340 degrees Fahr. (725 degrees Cent.), but their toughness is not so high at the low temperatures when the steel has been previously normalized at 1650 degrees Fahr. (900 degrees Cent.).

The low-carbon 3 per cent chromium steel has good fabricating properties. According to the experience on the small ingots it can be hot-rolled in substantially the same manner as ordinary carbon steels. Its high ductility makes it possible to bend or otherwise form sections of the steel either hot or cold into any article produced from ordinary carbon steel. The 3 per cent chromium steel can be welded; and the welds have good toughness at room temperature and at temperatures down to -40 degrees Fahr. (-40 degrees Cent.).

The utility of the low-carbon 3 per cent chromium steel becomes apparent when its good fabricating properties and resistance to atmospheric deterioration are jointly considered. Articles of the steel such as roofing, gutters, drain pipes, and other parts exposed to weathering will last for long periods. Fence wire represents another article to be made from the steel, as well as certain structural parts for applications in which atmospheric corrosion is a serious menace.

DISCUSSION

Written Discussion: By G. DeVries, assistant metallurgist, National Bureau of Standards, Washington, D. C.

In Fig. 6, the author has shown that the hardness of 1-inch rounds cooled from 1650 degrees Fahr. (900 degrees Cent.) in air increases markedly with chromium in excess of about 3 per cent. Since the cooling rates obtained by cooling in air will vary with the size of the specimens, it is to be expected that the location of the break in Curve 2 would change with the size of the specimen. Possibly its position would be shifted to the left with smaller and to the right with larger rounds than those used by the author. Would the increase in cooling rate, due to a change from 1 to $\frac{1}{4}$ -inch round or to some size normally used for cold drawing wire, be sufficient to cause air hardening of the 3 per cent chromium steel? If this steel does show an appreciable increase in hardness by cooling the smaller sections in air, then its use for the production of wire would result in some increase in cost over that of low-carbon steel due to the necessity for additional annealing or for the use of a higher powered cold drawing process.

Oral Discussion

W. P. WOOD:³ In the last photomicrograph that Mr. Franks presented, he called attention to a structure that he designated pseudomartensite. Was there any difference in the corrosion characteristics where such a structure existed as compared to the more or less spheroidized structure?

GLENN R. INGLES:⁴ Mr. Franks is to be congratulated for his very excellent paper.

I would like to ask one question and that is: What is the effect of nitrogen on the air hardening properties of the 3 per cent chromium steel?

N. A. ZIEGLER:⁵ Corrosion resistance of chromium steels is affected, to a considerable extent, by their structural characteristics developed by heat treating. It should be remembered that steels containing over 2 per cent chromium and 0.1 per cent carbon are more or less air hardening. This means that even on relatively slow cooling they pass through a suppressed transformation and possess martensitic or bainitic structures. Upon drawing, this structure is broken up into fine carbides dispersed in a ferritic background.

On the other hand, if one of these steels is austenitized and then cooled down and held at 1290 to 1330 degrees Fahr. (700 to 720 degrees Cent.), it passes through an isothermal transformation and results in a pearlito-ferritic structure.

We have no data on standard corrosion testing of steels thus treated, but we know from our metallographic experience that normalized and drawn, or quenched and drawn steels are much less susceptible to metallographic acid reagents' attack than similar steels isothermally treated. This seems to be an indication that the former are more "corrosion resistant" than the latter.

Author's Reply

The discussion by Mr. DeVries in which he raises the question of the effect of thickness of section on the air hardening characteristics of the low-carbon 3 per cent chromium steels is an extremely important one in view of the possible use of this steel in the form of fence wire. He has asked whether the increase in cooling rate brought about by changing the section from a 1-inch round bar to a 1/4-inch round rod would cause the metal to air harden appreciably on normalizing. The Rockwell B data of Table III obtained on normalizing 20-gage sheet of the low-carbon 3 per cent chromium steel are pertinent, as sheet of this thickness represents an extreme condition in regard to the cooling rate when heated at 1650 degrees Fahr. (900 degrees Cent.) and air-cooled. It is shown that sheet heated for 2 hours at 1340 degrees Fahr. (725 degrees Cent.) and air-cooled has a Rockwell B value of 66, whereas after heating for 10 minutes at 1650 degrees Fahr. (900 degrees Cent.) and air cooling the sheet has a Rockwell B value of only 75. This shows an increase in hardness over the fully softened sheet, but it is clear that the metal is still in a soft state. Other data on 1/8-inch thick sheet and 1/4-inch thick plate show that the low-

³Professor of metallurgical engineering, University of Michigan, Ann Arbor, Mich.

⁴Metallurgical consultant, Cook Heat Treating Co., Houston, Texas.

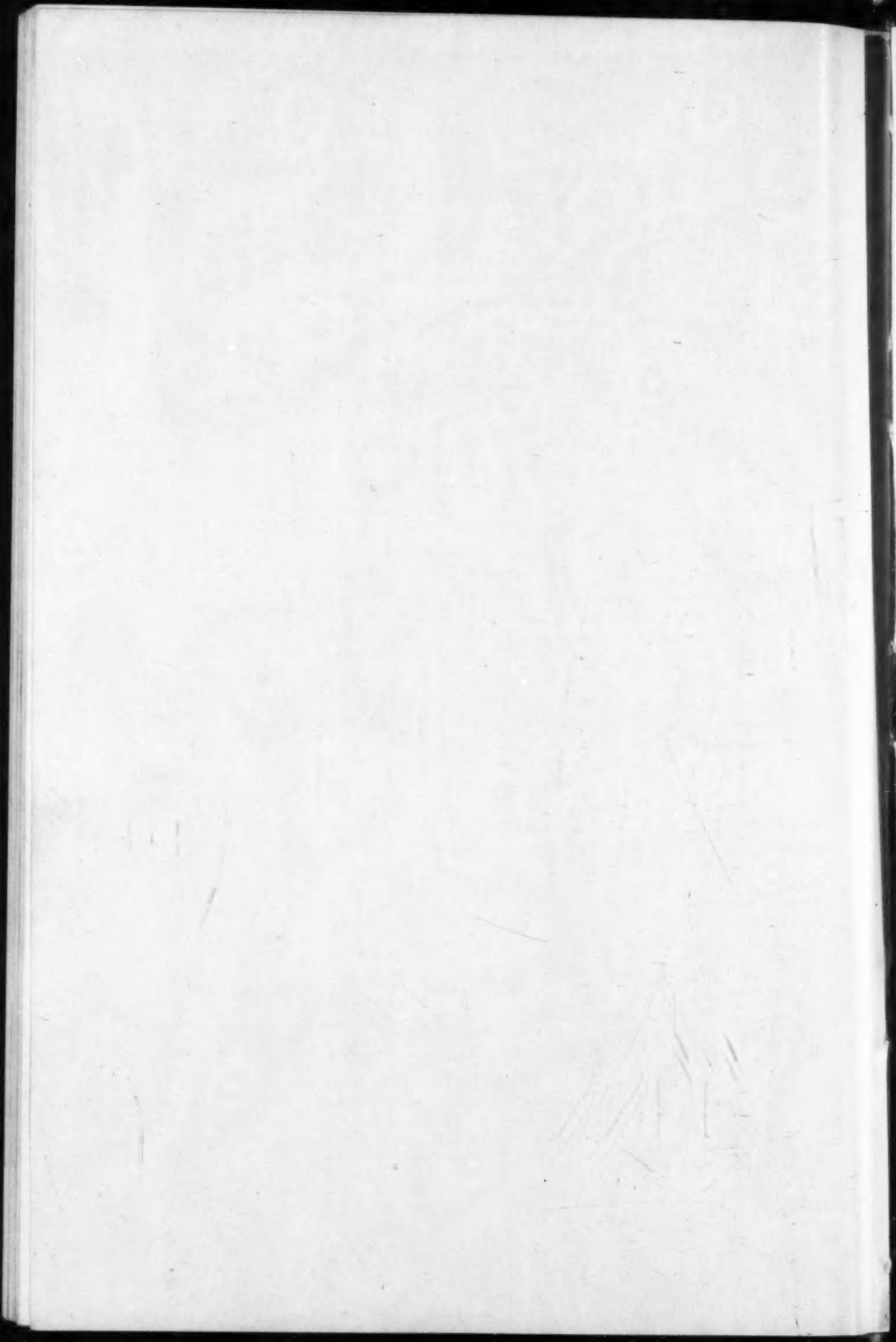
⁵Research metallurgist, Crane Co., Chicago.

carbon 3 per cent chromium steel will not greatly harden if it is air-cooled from about 1650 degrees Fahr. (900 degrees Cent.). It is therefore our opinion that wire of the low-carbon 3 per cent chromium steel can be air-cooled from 1650 degrees Fahr. (900 degrees Cent.) without causing the metal to seriously air harden.

Mr. Ingles has raised a question in regard to the effect of nitrogen on the air hardening properties of the low-carbon 3 per cent chromium steel. Our experience shows that the maximum percentage of nitrogen that can be retained in stable combination in high chromium steels is approximately one-hundredth of the chromium content. This means that the low-carbon 3 per cent chromium steel could retain as much as 0.03 per cent nitrogen, which in our opinion would cause the steel to become more subject to air hardening than if the nitrogen is held, as in the present steels, in the range 0.01 to 0.015 per cent. It is not believed that the presence of 0.03 per cent nitrogen would cause these steels to harden to such an extent that the metal would become relatively brittle. The effect of nitrogen is to harden the high-chromium steels without greatly reducing their toughness.

Mr. Ziegler's comments on the effect of structure on the corrosion resistance of the steels is in accordance with our experience. He states that in general the high-chromium steels are more resistant to corrosion in the normalized and drawn, or quenched and drawn condition than they are in the fully softened condition. The structural effect on the corrosion resistance of the high-chromium steels is also greatly influenced by the percentage of carbon the steels contain. When the carbon content is high, the difference between the corrosion resistance of the normalized and drawn, or quenched and drawn steels, and that of the fully softened steels is quite appreciable. On the other hand, if the carbon content is low, the difference in the corrosion resistance after the respective heat treatments is nowhere nearly as great. This is well illustrated by work done on the cutlery type of stainless steels, and on the lower carbon stainless steels of approximately the same chromium content known as the rustless irons. Thus, it can be stated, in answer to Professor Wood's question, that the corrosion resistance of the low-carbon 3 per cent chromium steels would not be greatly influenced by changes in structure resulting from different heat treatments.





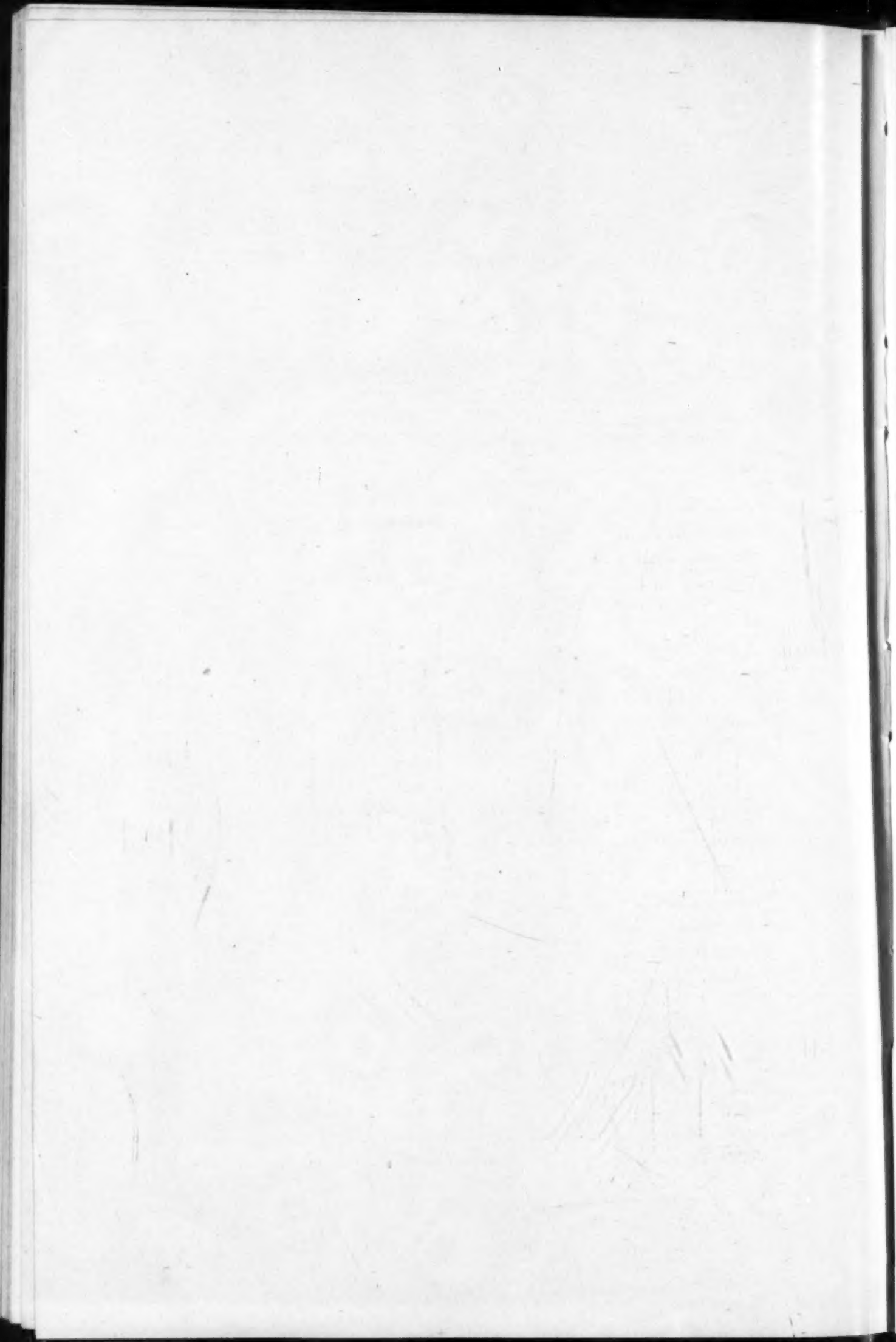
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